CYCLONE ENGINEERING
SALES LTD.

95 pp

MICROMEDIA

CONTRACT SERIAL NUMBER 69139-01-SQ

DSS REQUISITION NUMBER 23440-6-9139

CONTRACTOR: CYCLONE ENGINEERING SALES

FINAL REPORT
Prepared for
DSS 23440-6-9139101ISQ
by
Waterloo Centre For Process Development
(Plastofrost Measurements)

CYCLONE ENGINEERING SALES LTD.
9719 - 60 Avenue
Edmonton, Alberta
T6E 0C4

Telephone: (403)436-1385
Telex: 037-3793
Fax: (403)436-4103

W.C.P.D. University of Waterloo

Waterloo Centre for Process Development

FINAL REPORT

Prepared for

CYCLONE ENGINEERING SALES LIMITED

Plastofrost Measurements

Principal Investigator: Dr. P.L. Silveston

Research Engineer: A. Royce

January 1988

TABLE OF CONTENTS

	PAGE
SUMMARY	1
CONCLUSIONS	2
RECOMMENDATIONS	4
INTRODUCTION	6
Plastofrost Carbonization	7
EXPERIMENTAL EQUIPMENT AND PROCEDURES	8
Description of Equipment	8
Feedstocks	11
Plastofrost Coal Run Procedure	11
Plastofrost Slurry Run Procedure	16
Microscopic Examination	19
RESULTS AND DISCUSSION	22
Heating Profiles	22
Microscopic Investigations	32
REFERENCES	71
APPENDICES	
Appendix A: Eequipment and Procedure Development	72
Appendix B: Description of Plastofrost Samples	75
Appendix C: Significance of Optical Texture of Carbonized Coal	80
Appendix D: Work Statement	84

LIST OF FIGURES

			PAGE
FIGURE	1	Modified Plastofrost Apparatus	9
	2A	Arrangement of Plastofrost Unit in Fume Hood and Location of Microcomputer Controller and Data Logger	10
	2B	Plastofrost Unit - Furnace with Insulation	10
	3A	Coking Attachment for Coal Samples	12
	3B	Coking Attachment for Slurries	12
	4	Schematic of Vacuum Impregnation Unit	15
	5A	Buehler Polishing Bench	17
	5B	Photographs of Polished and Unpolished Sections	17
	6	Heating Profile - PRC5	27
	7	Heating Profile - PRN-30	28
	8	Heating Profile - LNN-30B	29
	9	Heating Profile - LNN-30C	30
	10	Heating Profile - PRC20-30	31
	11	Photomicrograph of Forestburg Coal	38
	12	Photomicrograph of Forestburg Coal Slurry	41
	13	Photomicrograph of LHOP	44
	14	Photomicrograph of Prince Mine Coal	47
	15	Photomicrograph of Lingan Mine Coal	50
	16	Photomicrograph of Lingan Mine LNC20 and LNC5 Coals	52
	17	Photomicrograph of Prince Mine PRC20 Coal	57
	18	Photomicrograph of Prince Mine Coal Slurries	59
	19	Photomicrograph of Lingan Mine Coal Slurries	62

PAGE

FIGURE	20	Photomicrograph of Lingan Mine LNN-30A and LNN-30C Coal Slurries	65
	21	Photomicrograph of Lingan Mine LNC5-30 Coal Slurries	67
	22	Photomicrograph of Lingan Mine LNC20-30 Coal Slurries	68
	23	TGA Plot for LHOP	70A

LIST OF TABLES

			PAGE
TABLE	1	Coal and Slurry Feedstocks	13
	2	Coal Plastofrost Final Temperatures	23
	3	Coal-Oil Slurry Plastofrost Final Temperatures	24
	4	Classification of the Optical Texture in Cokes	33
	5	Results from Microscopic Examination of Forestburg Plastofrost Samples	34
	6	Results from Microscopic Examination of Illinois #6 Plastofrost Samples	35
	7	Results from Microscopic Examination of Prince Plastofrost Samples	36
	8	Results from Microscopic Examination of Lingan Plastofrost Samples	37

SUMMARY

The objective of this study was to investigate the inhibition of coke fusion and/or formation by the ferrous sulphate additive employed in the CANMET process of bitumen-coal coprocessing. A Plastofrost apparatus was used for the above purpose, along with a specially developed procedure to handle the slurry. Three Canadian coals - Forestburg (sub b-C), Prince Mine (hvb-B), Lingan Mine (hvb-A) were used along with an Illinois #6 (hvb-C) coal. The slurry fluid was a Cold Lake vacuum bottoms. Plastofrost samples were prepared of the coals with 0, 5 and 20 wt% additive, the vacuum bottoms, and slurries of 30 wt% coal in the vacuum bottoms with additive levels of 0, 5 and 20 wt %. Prince Mine and Illinois #6 coals did not become fluid suggesting ageing of the samples had occurred. With the Lingan Mine coal, additive delayed initial fusion of the coal grains and completion of fusion. At 20 wt% additive complete fusion did not occur. The anisotropic texture of the semicoke was diminished by the additive. The presence of vacuum bottoms suppresses coal fusion, probably by physically separating the coal grains. Fusion is also slightly suppressed at 20 wt% additive; 5 wt% seemed to have little effect. Dissolution of vitrain in the bitumen was not observed. The coal and vacuum bottom phases carbonize separately yielding distinct, but well bonded semicokes. Anisotropic texture of the vacuum bottoms coke is strongly diminished by the presence of the finely ground coal.

CONCLUSIONS

- Initial fusion of coal grains and the disappearance of grain boundaries (completion of fusion) are both delayed to higher temperatures with increasing levels of additive (ferrous sulphate) in the coal. Complete fusion does not occur (by 560°C) with 20 wt% additive.

- The presence of additive (5 wt% and 20 wt%) diminished the anisotropic texture of the coal.

- No effect of the additive was observed with the non-caking coal or caking coals which had aged prior to our tests.

- The presence of CLVB alone decreases the extent of coal fusion although this may be due to the lower packing density of the coal particles (i.e., coal grains are prevented from fusing together because they are separated by CLVB).

- The fusion of coal grains in the slurry sample is slightly inhibited by the additive with 20 wt% ferrous sulphate and negligibly affected by the additive with 5 wt% ferrous sulphate.

- There is no dissolution of the coal/vitrinite grain in the CLVB; each phase carbonizes separately yielding distinct cokes. However, the anisotopic texture of the CLVB is strongly diminished by the presence of coal and this effect is stronger for non-coking coals.

- The very small particle size (-75 microns) of the coal samples may have

accelerated the effects of aging on the coking properties of the Prince and Illinois samples. Our tests showed they behaved as non-caking coals.

- Duplicate Plastofrost tests done with Lingan coal indicate that some oxidation of the sample had probably occurred.

- No effects of coal aging were observed with the slurry samples.

- The Plastofrost method is an excellent device for examining the effects of additives and sample aging on the coking properties of coal.

- Despite extensive modifications, the Plastofrost method did not provide a fully satisfactory method for studying coke formation in slurry samples. Further development is needed to eliminate mixing of the bitumen and separation of the coal (see Recommendations). With this accomplished, the technique is potentially very useful.

RECOMMENDATIONS

1. Determine the optimal level of $FeSO_4$ and/or K_2CO_3 required to inhibit fusion of coking coals. To be relevant to coprocessing, this should be checked in a H_2 atmosphere.

2. Plastofrost is best suited to the study the transformation of coal or residue to coke, therefore, work should be limited to determining the effect of additives and/or aging on the coking properties of coal or of mixtures of coal and petroleum residues which do not become fluid in the coking cylinder.

   Possibly, if the bitumen fraction of the coprocessing slurry was distilled (carbonized) prior to a Plastofrost run, the temperature gradients normally observed in coal runs would be present and the coked residue or coal/residue would provide visualization of the transformation to coke with increasing temperature which is needed to understand the solidification process.

3. The co-processing residue may be a feasible alternative source of metallurgical coke or it may serve as a coke blend. A strong material seems to be formed by coking.

4. For coal studies, larger particle sizes, i.e. 850 microns (20 mesh), are recommended to facilitate identification of macerals (by structure). Particle size may be an interesting parameter in a study on the effect of additives - other parameters such as heating rate, atmosphere, and other

ranks of coal should also be examined.

INTRODUCTION

The Synthetic Fuels Research Laboratory of CANMET is involved in an extensive R & D program to simultaneously liquefy coal and upgrade heavy oil. This coprocessing is accomplished in a single-stage, once-through operation which combines aspects of both coal liquefaction and heavy oil hydrocracking technologies. It utilizes an iron-based disposable additive supported on coal. It was shown that the process is feasible for low-rank Western Canadian coals, however, some Eastern Canadian high-volatile bituminous coals resulted in severe plugging problems due to the coal's swelling and thermoplastic properties. Increasing the concentration of the CANMET additive by impregnating all the coal with 10-20% iron sulphate made it possible to process the bituminous coals without flow problems[1].

The objective of this project was to investigate the inhibition of coke formation by the CANMET additive used in coprocessing and elucidate the mechanism involved as far as possible. Use has been made of the Plastofrost apparatus, which essentially acts as a small coke oven, but with the sample heated from just one side. The apparatus is capable of producing samples in which the carbonization process can be visually inspected. The "Work Statement" asked the Waterloo Coal Research Laboratory to examine 24 samples produced with the plastofrost apparatus from coals of varying rank and additive level and from slurries of these coals in heavy oil.

Plastofrost Carbonization

The Plastofrost was developed by Ritter and Juranek of West Germany in 1960[2]. Their aim was to obtain photo-micrographs of the coal-to-coke transformation, permitting a study of the softening process and the interaction of coals at different degrees of carbonization. The Plastofrost shows the changes occurring at different temperatures during the process side by side, thus producing a time-temperature display of the softening process. To achieve this, it was necessary to produce a temperature difference within the sample extending from 350°C to about 550°C. Also, the sample had to be large enough to render all changes visible, but small enough to allow a good polished section to be produced.

In a coke oven, heat is transmitted from the walls to the centre of the oven, and ideally, isothermal surfaces are approximately parallel to the walls. To obtain analogous heating in a small test sample, unidirectional heating must be used. The plastic zone would then initially form at the face in contact with the heated surface and move as a front through the coal mass to the opposite face. Abramski and Mackowski found that the essential phases of coke formation occurred in the plastic zone and that the optical texture of coke does not alter significantly on heating up to 1500-1700°C. Consequently, high temperatures are unnecessary. A maximum temperature of about 550°C to ensure that semi-coke forms would suffice for observation of coke texture.

EXPERIMENTAL EQUIPMENT AND PROCEDURES

Description of Equipment

In preparation for this work some equipment modifications were made and these are discussed in Appendix A. Figure 1 illustrates the Plastofrost apparatus used in the present study[3]. The two main components are the furnace and the coking attachment. The furnace consists of a nickel-plated copper slab in which four 300 watt cartridge heaters are enclosed. A chromel/alumel thermocouple placed beneath the top surface of the slab measures the temperature. The coking attachment is made of 316 stainless steel and comprises three parts: the baseplate; the coking cylinder; and the ram. The coking cylinder has an inside diameter of 41mm and a height of 75mm. It can be separated vertically into halves and has ten 2.5mm diameter holes spaced 5mm apart centred along each side of the vertical split. Ceramic tubes (2.49mm o.d, 1.57mm i.d.) are placed in these holes, as shown in Figure 1, and during a plastofrost run 1/25" diameter chromel/alumel thermocouples are placed in the tubes so that the measuring junctions are located along the axis of the cylinder. The coking attachment is placed on the furnace which provides unidirectional heating to the coal in the cylinder from the bottom upwards. A microcomputer based control and data acquisition system is required to monitor the 10 thermocouples in the sample and to monitor and control the furnace temperature. Figure 2-A shows the plastofrost apparatus in the fume hood accompanied by the computer and printer. Figure 2-B shows the coking attachment with the thermocouples inserted prior to being insulated and placed on the furnace.

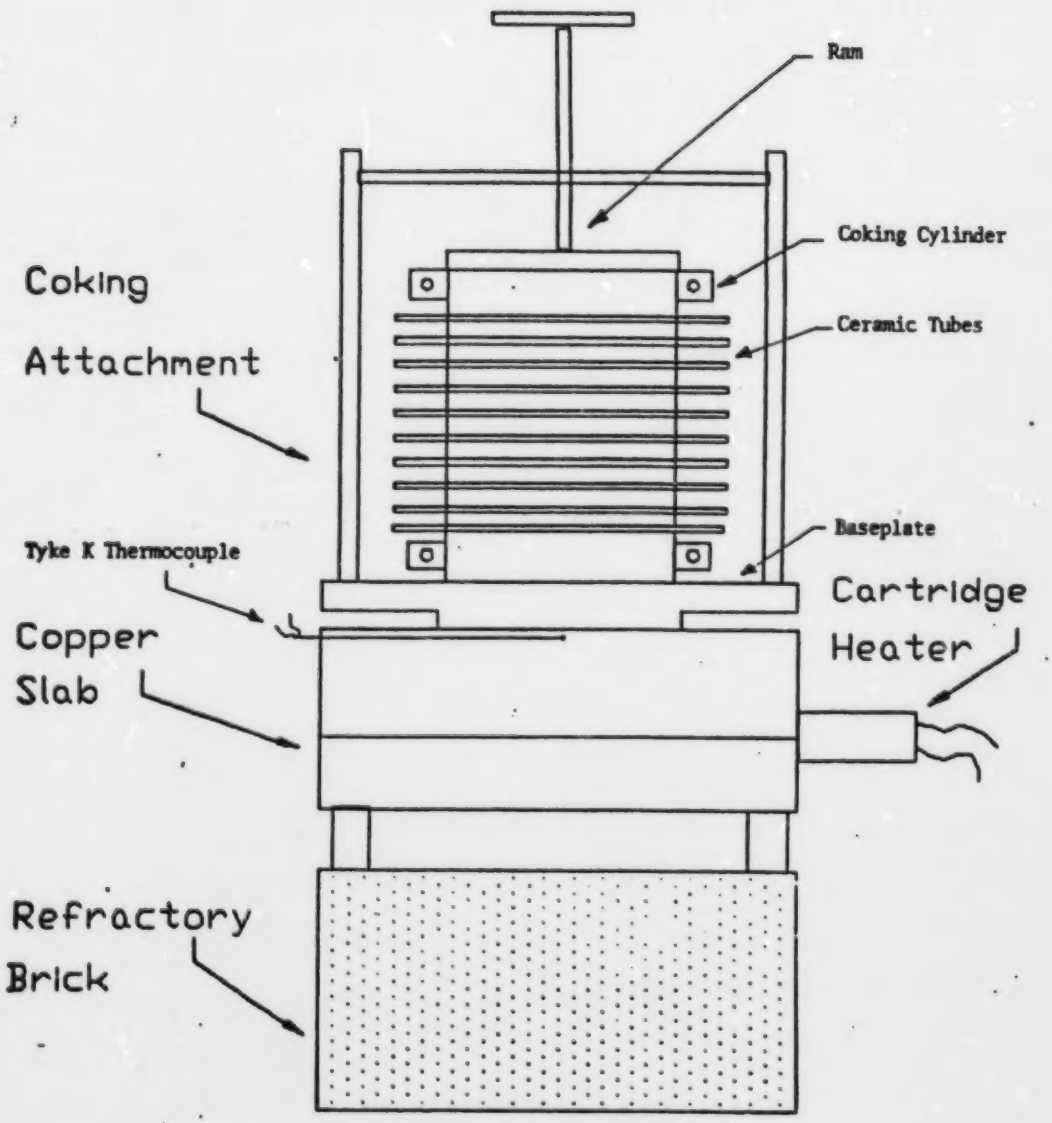
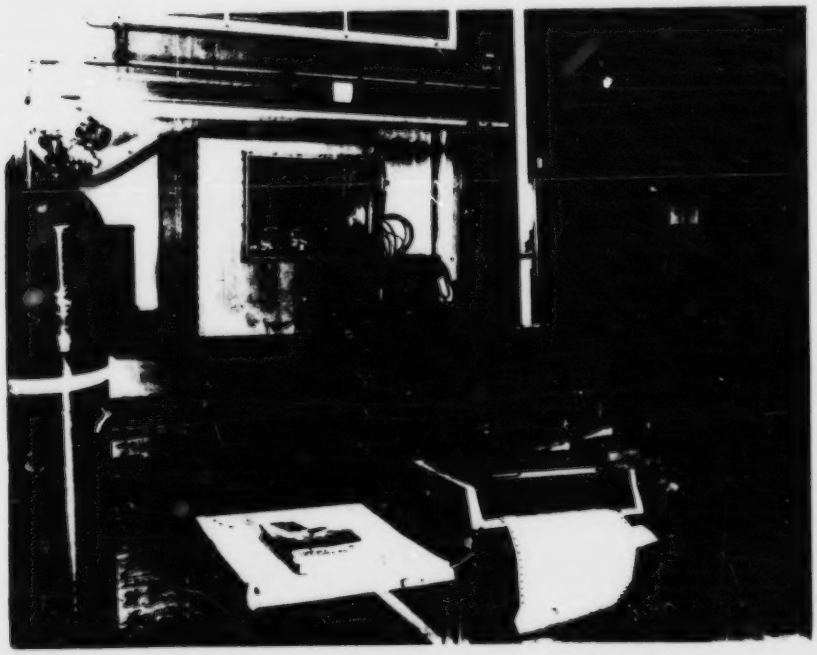
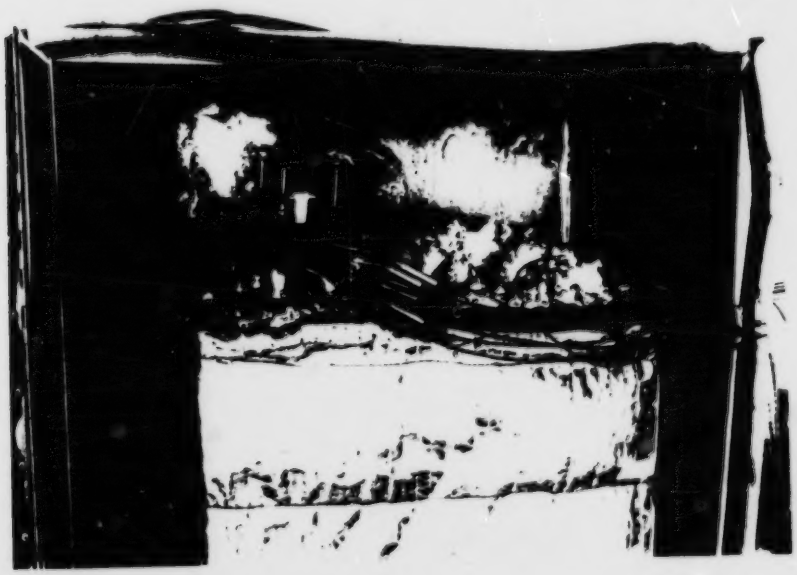


FIGURE 1: Modified Plastofrost Apparatus

Figure 2: Plastofrost Unit Arrangement



A. Arrangement in Fume Hood with Location of Microcomputer Controller and Data Logger



B. Plastofrost Unit and Furnace with Insulation

Feedstocks

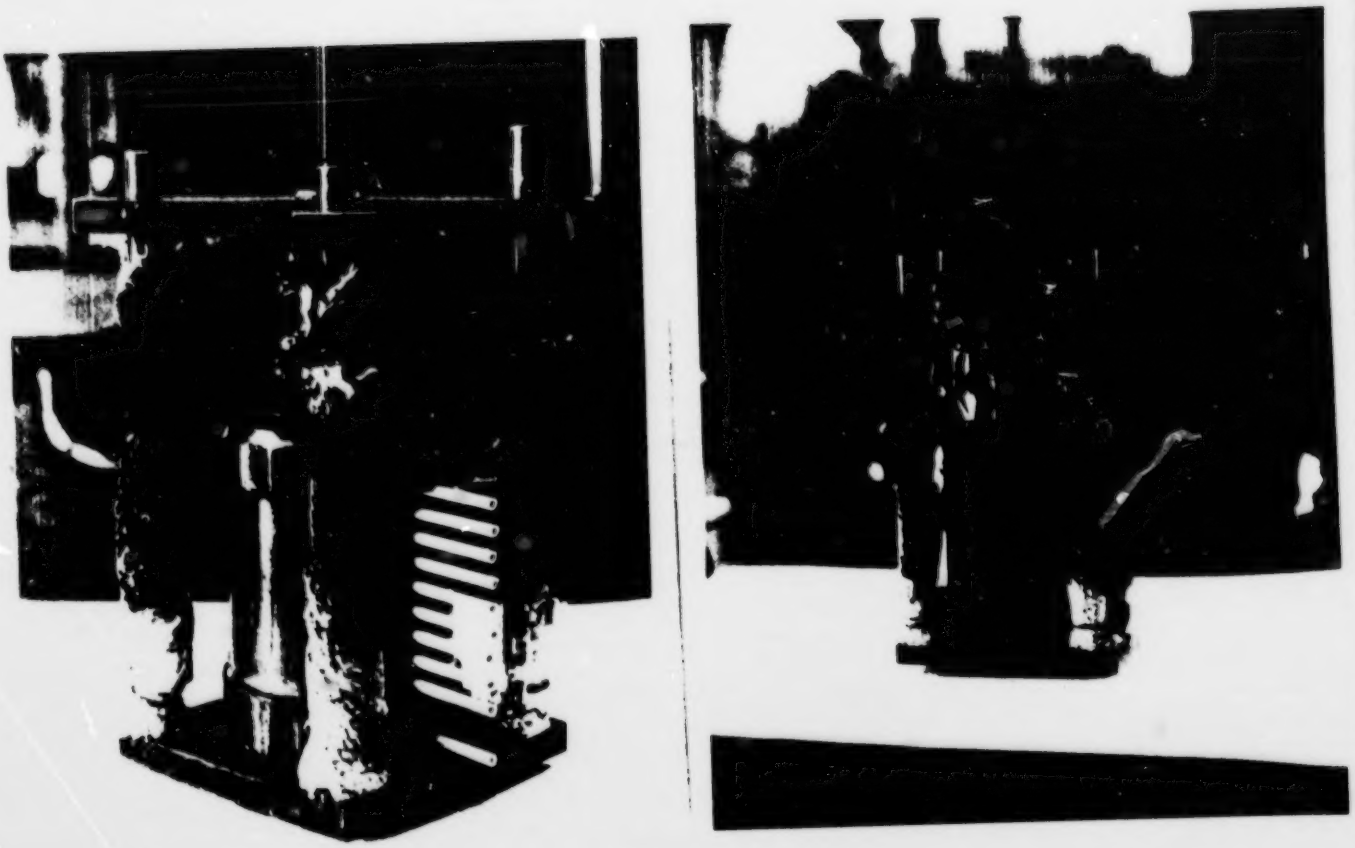
Four different coal feedstocks were tested: Illinois #6, a U.S. hvbC; Prince mine, a hvbB, and Lingan mine, a hvbA, both from Eastern Canada: and Forestburg, a sub-bituminous C from Alberta. Each coal was previously treated with the CANMET additive by impregnation with 5 and 20 wt% ferrous sulphate heptahydrate using a water slurry. The coals had also been ground and sized to minus 200 mesh (-75 micron). Thus 12 samples were tested consisting of 0, 5 and 20 wt% iron sulphate. Twelve slurry samples were also tested which contained 30 wt% coal in Cold Lake vacuum bottoms (CLVB) for each coal sample described above. Table 1 lists the coal and slurry samples used.

Plastofrost Coal Run Procedure

A sample of as-received coal, ground to -200 mesh, weighing 66.5g, is incrementally packed into the coking cylinder which is lined with heavy gauge aluminum foil. Ceramic tubes are arranged in the coking cylinder, as shown in Figure 3-A, prior to the addition and packing of the coal. The ram is adjusted to guarantee a constant packing density of 7.2 g/cm^3 for all samples. The apparatus is then placed on the furnace plate, the thermocouples are inserted into the ceramic tubes, and the furnace and coking attachment are insulated to ensure that a 200°C temperature drop is obtained within the sample.

The samples are heated in air until the temperature at the lowest level in the coal reaches 200°C. At this time, nitrogen is introduced into the test

Figure 3: Coking Attachment for Plastofrost



A. Attachment for Coal Samples showing T/C Locations

B. Attachment for Coal Slurries showing Aluminum Foil Extension

TABLE 1: Coal and Slurry Feedstock

Sample Code	Coal Name	Coal Rank	Weight Percent Additive in Coal	Weight Percent Coal in Slurry
FBN	Forestburg	Sub-bituminous	0	
FBC5			5	
FBC20			20	
ILLN	Illinois #6	High-Volatile	0	
ILLC5		Bituminous C	5	
ILLC20			20	
PRN	Prince	High-Volatile	0	
PRC5		Bituminous B	5	
			20	
LNN	Lingan	High-Volatile	0	
LNC		Bituminous A	5	
			20	
FBN-30	Forestburg	Sub-bituminous	0	30
FBC5-30			5	30
FBC20-30			20	30
ILLN-30	Illinois #6	hvb C	0	30
			5	30
			20	30
PRN-30	Prince	hvb B	0	30
PRC5-30			5	30
PRC20-30			20	30
LNN-30	Lingan	hvb A	0	30
LNC5-30			5	30
LNC20-30			20	30

chamber. The furnace is heated at its maximum rate until its temperature reaches 400°C at which time the temperature at the lowest level in the coal is still about 200°C. Above 400°C the furnace heating rate is controlled at 3°C/min. As soon as the temperature at the sample bottom reaches about 550°C, heating is stopped and the coking attachment is quickly removed from the furnace and left to cool in a nitrogen atmosphere. At this time point, the temperature at the sample top should not have risen above 350°C.

After cooling, the charge, together with the ceramic tubes, is impregnated with polyester resin. Once the resin hardens, the impregnated sample is cut into halves along a plane perpendicular to the ceramic tubes.

To ensure complete saturation of the exposed surface with resin a vacuum impregnation technique described by Duever[3] is used to force the resin into the voids. Figure 4 shows a sketch of the vacuum impregnation apparatus used in this study.

The sample is placed in an aluminum foil mould, measuring approximately 4.5 cm x 7.75 cm with the exposed face upward. It is then placed in the vacuum desicator and degassed for 5 minutes while the resin is being mixed. The resin is then introduced into the sample through a teflon tube and the flow is adjusted with a supply clamp. Since air is introduced with the resin, the sample is degassed for an additional 5 minutes. Valve B is closed thereby isolating the pump and valve A, which was closed up to this point, is opened. The pressurization of the desicator provides the "push" to force the resin into the pores.

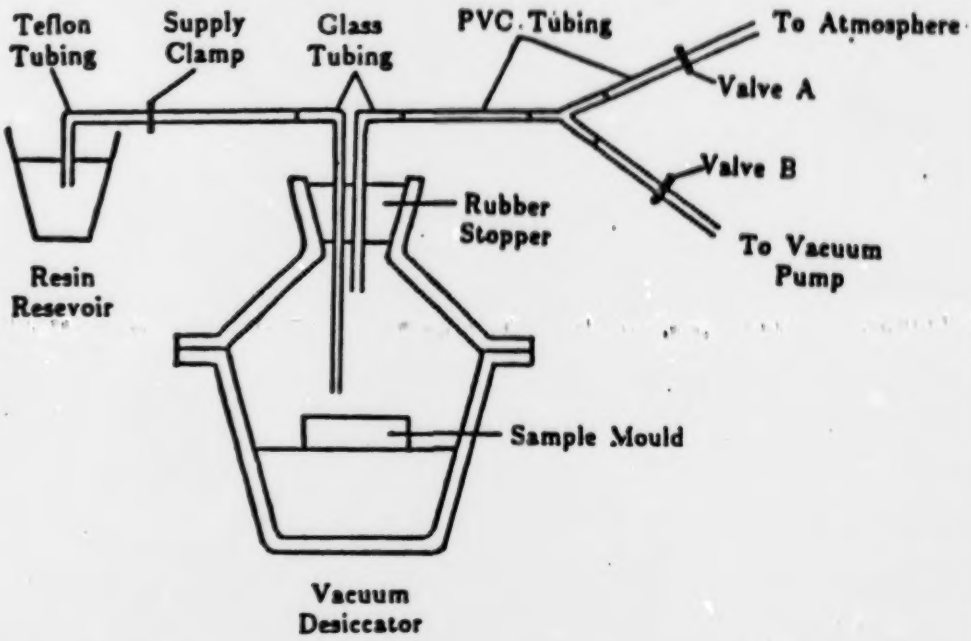


Figure 4: Schematic of Vacuum Impregnation Apparatus

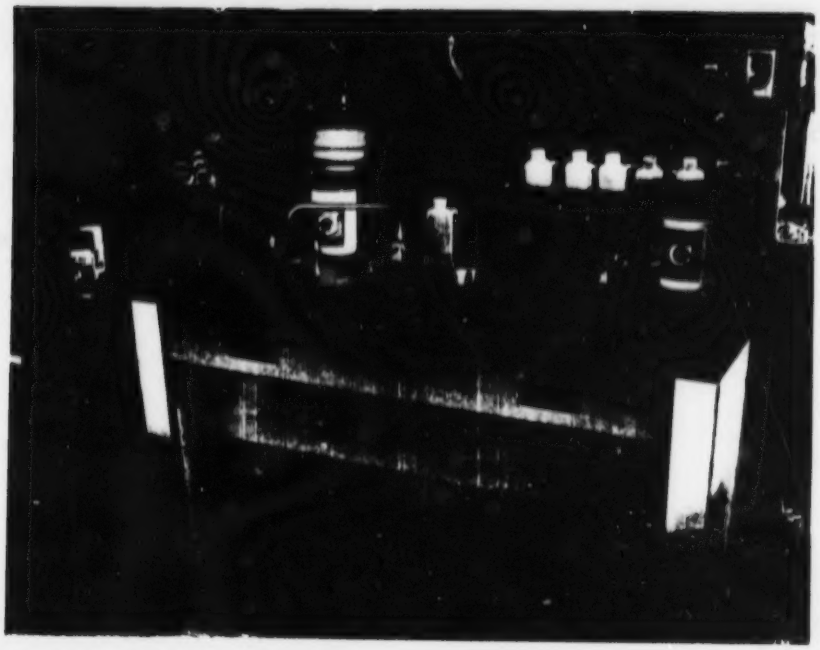
The hardened samples are ground in three stages using silicon carbide abrasive papers (240, 400, 600 grit) mounted on an 8" bronze grinding wheel. The grinding and polishing bench is shown in Figure 5-A. Methanol or 2-propanol was used as a coolant instead of water to prevent loss of iron from the treated samples. Polishing is also done in three stages using alumina polishing abrasives (1, 0.3, 0.05 microns) slurried in alcohol. The samples are thoroughly cleaned in alcohol in an ultrasonic cleaner between each stage. Polished and unpolished plastofrost samples are shown in Figure 5-B.

Plastofrost Slurry Run Procedure

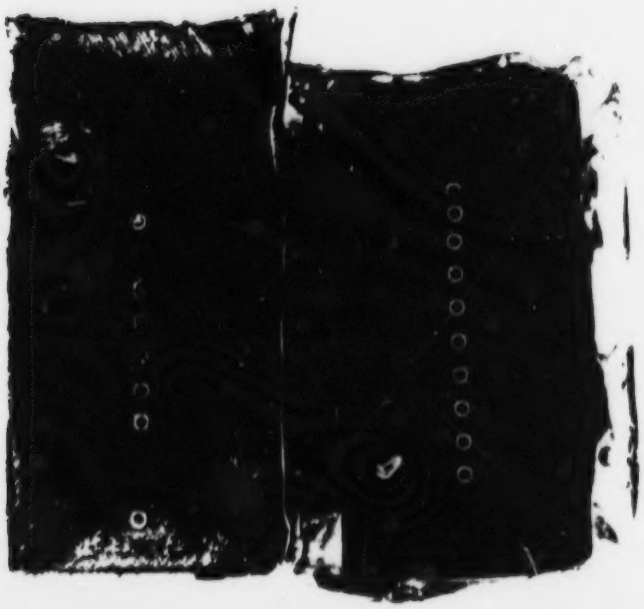
Because of the fluidity when the slurry is heated, a new technique for handling these samples had to be devised. The development of this technique is discussed in Appendix A. In this we describe only the final method arrived at.

Before charging the coking cylinder with a slurry sample, the cylinder, together with the aluminum lining, is sealed with a water glass (sodium silicate) solution. To increase the height of the cylinder, the aluminum lining is extended about 6 cm above the top of the cylinder as shown in Figure 3-B. These procedures prevent the hot bitumen from boiling over or leaking out of the cylidner. To take a representative sample of a slurry for an experiment, the entire sample received from CANMET is heated in an oven at 105°C for approximately two hours. The fluid slurry is then stirred well and approximately 70g transferred into the coking cylinder. For experiments with slurries, the ram attachment is not necessary and was not used. The coking attachment (cylinder and baseplate) is then placed on the furnace plate,

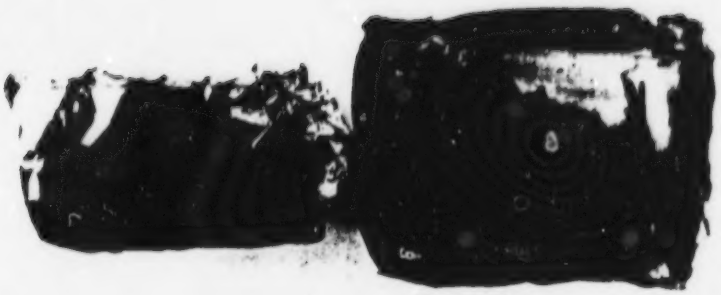
Figure 5: Polishing Arrangement and Polished Samples



A. Buehler Polishing Bench



B. Unpolished Sample (on left) with Resin which did not Percolate through sample (on top) and Polished Sample (on right) with Excess Resin



C. Polished Samples from Coal Slurry Runs showing Excess Resin

thermocouples inserted, and the cylinder insulated.

For most Plastofrost slurry runs, the furnace is heated at its maximum rate to 400°C and held at that temperature. Nitrogen is introduced before the sample reaches 200°C. The furnace is controlled so that the sample approaches its boiling point (~400°C) gradually. When boiling has ceased the sample is heated to the final run temperature and held there for 10 to 180 minutes. The coking attachment is then removed from the heat and allowed to cool in a nitrogen atmosphere.

Four slurry runs employed non-standard temperature programs in which the sample is held at temperatures from 350°C to 520°C overnight before heating to the final temperature. One sample was heated to boiling, cooled before boiling ceased, and reheated the following day to the boiling point, and then to the final temperature when boiling stopped.

Preparation for microscopic examination is done following the same procedures used for coal samples.

The procedures for testing the CLVB sample were the same as the slurry test procedures.

Microscopic Examination

The microscope used in this study is a Zeiss Large UNIVERSAL Research

Microscope. The polished plastofrost samples are examined by reflected light using parallel polarizers and a 1/4 plate inserted between the specimen surface and the analyzer to characterize the samples in terms of their optical texture.

The various samples are examined to observe the softening characteritics of the coal, resolidification of the coal and heavy oil, and the nature of the coke formed. The CANMET additive is also characterized according to its distribution in the samples. Specifically, coal plastofrost samples were examined to determine the temperatures corresponding to the following changes:

1. The first appearance of pores in the individual grains.
2. The initial fusion of the grains.
3. The point at which there is a significant increase in the proportion of pores.
4. The completion of fusion where individual grains are no longer distinguishable.
5. The development of anisotropy

Most observations are made at 500x magnification in oil in order to distinguish the very fine grains (-200 mesh) of coal and the CANMET additive. Lower magnifications (50x, 100x and 200x) are primarily used to determine the overall structure of the coke formed in terms of porosity and extent of fusion.

Coked slurry samples did not have a large temperature gradient within the samples and their examination, therefore, differed from that of the coal samples. Lower magnification in air (50-200x) were used to characterize the

larger structural features of the sample. The individual coal grains, the CANMET additive and the optical texture were best observed at magnifications of 500x in oil.

Interpretation of the features requires skill and experience even though the visual appearance is considerably better than indicated by the photographs which accompanies the Results and Discussion section. A thorough literature review was undertaken to understand semi-coke anisotropy. The significance of coke optical texture is discussed in Appendix C.

RESULTS AND DISCUSSION

Heating Profiles

Fourteen plastofrost runs were made with the 12 coal samples and 17 runs were made with the slurry samples. Two runs were also made with the CLVB sample. The final temperature data for the coal runs are given in Table 2. These data represent the maximum temperatures reached at each of the thermocouple locations. Temperature data for the slurry and CLVB runs are summarized in Table 3. Some of these runs included a soak period at an intermediate temperature before heating to the final temperature. Heating profiles for some of these runs are shown in Figures 6 to 10. Figure 6 illustrates the typical heating profile for a coal run in which the heating rate is maintained at approximately 3°C/min and a final temperature drop within the sample of 200°C, from 550°C to 350°C is achieved. Figure 6 does not show the initial heating of the sample and furnace from room temperature. As previously described, the furnace is initially heated at its maximum rate (~10°C/min) until it reaches 400C. At this point the furnace power is adjusted to maintain this temperature until the bottom thermocouple in the coal reaches about 200°C. This is shown in Figure 6. The furnace is then heated at 3°C/min until the final temperatures are reached. In all of the coal runs, samples were heated in this manner.

It was learned from initial slurry runs that the hot CLVB was mixing in the coking cylinder during heating and that temperature gradients were absent until resolidification occurred. Furthermore, the loss of volatiles resulted in the final coke residue occupying only the bottom third of the coking cylinder.

TABLE 2: Coal Plastofrost Final Temperatures

Sample Name	Date Run	T_1	T_2	T_3	T_4	T_5	T_6	T_7	T_8	T_9	T_{10}
FBN	Aug 21/87	561	514		460	437	417	398	373	353	328
FBC5	Aug 19/87	567	517	488	467	441	417	395	376	358	330
FBC20	Aug 4/87	555	517	496	475	452		402		360	341
ILLNA	May 22/87	552	529	508	488	467	445	424	408		374
ILLNB	June 26/87	544	504	487	468	450	430	411	395	377	360
ILLC5	Aug 12/87	570	524	500	479	455	429	406	386	363	344
ILLC20	Aug 18/87	557	518	498	474	449	424	402	382	358	337
PRN	June 25/87	541	498	483	463	441	423	406	387	370	353
PRC5	Aug 14/87	566	525	501	480	456	433	411	395	370	353
PRC20	Aug 10/87	565	521	500	480	457	433	409	389	368	348
LNNA	May 29/87	550	530	507	484	462	438	413	395		367
LNNB	June 25/87	580	511	494	470	450	426	406	388	367	349
LNC5	June 26/87	591	516	498	476	455	433	414	395	373	351
LNC20	July 21/87	560	500	460	436	412	388	369	350	328	

TABLE 3: Coal Oil Slurry Plastofrost Final Temperatures

Sample Name	Run Date	Soak Time (hrs)	Soak Temp (°C)	Final Temperature T_f (°C) Bottom —— Top	Time at T_f (hrs)
FRN-30	Oct 7/87			586, (422)*	.4
FBC5-30	Sep 15/87				.25
FBC20-30	Sep 29/87			569, 440	1.3
ILLN-30	Oct 5/87			550	0.7
ILLC5-30A	Aug 5-6/87	17	460	552, 473	0.5
ILLC5-30B	Aug 11/87			558	0.33
ILLC5-30C	Aug 7-8/87	7	520	650	0.15
ILLC20-30	Aug 13/87			562, (517)	0.4
PRN-30A	June 30/87			408	0.25
PRN-30B	July 17/87			508, 468, 446, 431, 411	0.5
PRC5-30	Sep 22/87			588	0.25
LNN-30A	July 28-29/87	17	250	460	0.5
LNN-30B	July 23&24/87	75	440	554, (490), (446)	0.25
PRC5-30	Sep 11/87			559	0.66
PRC20-30	Sep 22/87			588	0.25
LNN-30A	July 28-29/87	17	350	460	0.5
LNN-30B	July 23&24/87	.75	440	554, (490), (446)	0.25
LNN-30C	Sep 24/87	10	480	651	3
LNC5-30	Sep 24/87			567	2
LNC20-30	Aug 20/87			552	0.5
LHOP A	Oct 1/87				
LHOP B	Oct 9/87				

*Values in brackets indicate that the T/L was not fully immersed in the sample.

Therefore, the thermocouples in the top sections of the cylinder were unnecessary. Figure 7 shows the heating profile of one of these initial slurry runs. It is observed that temperature gradients were absent in the sample once the bitumen had risen above about 100°C and become very fluid. Gradients reappeared when boiling had ceased and the temperature had risen above about 420°C. At this point the sample volume was much less than the original volume and only the bottom 2 or 3 thermocouples accurately indicated the sample temperature.

Plastofrost sample LNN-30'B' was heated to boiling, cooled, and reheated the following day to 550°C. The heating profile for this run is shown in Figure 8. This sample (LNN-30B) was heated too quickly on the first day resulting in a boil-over when the thermocouple at the sample bottom was indicating 448°C. When this occurred the furnace power was turned off and the apparatus allowed to cool overnight. The following morning the spill was cleaned and the partly reduced sample was quickly reheated to boiling and held until boiling was complete before heating to the final temperature of 550°C.

Another Lingan slurry sample LNN-30'C' was held at 475°C for 12 hours and subsequently heated to 650°C and held for 3 hours. The temperature profile of this run is shown in Figure 9.

The temperature profile of a typical slurry run is shown in Figure 10. This plot illustrates the initial rapid heating to approximately 300°C followed by a more gradual heating to the initial slurry boiling point around 400°C. The temperature remained essentially constant while boiling progressed. Once boiling was complete the residue was heated to a final temperature of 588°C

and held for about 15 minutes. Loss of volatiles had exposed the upper thermocouple to the atmosphere and therefore it did not experience the final temperature rise. For other slurry runs the final temperatures varied from about 400°C to 650°C.

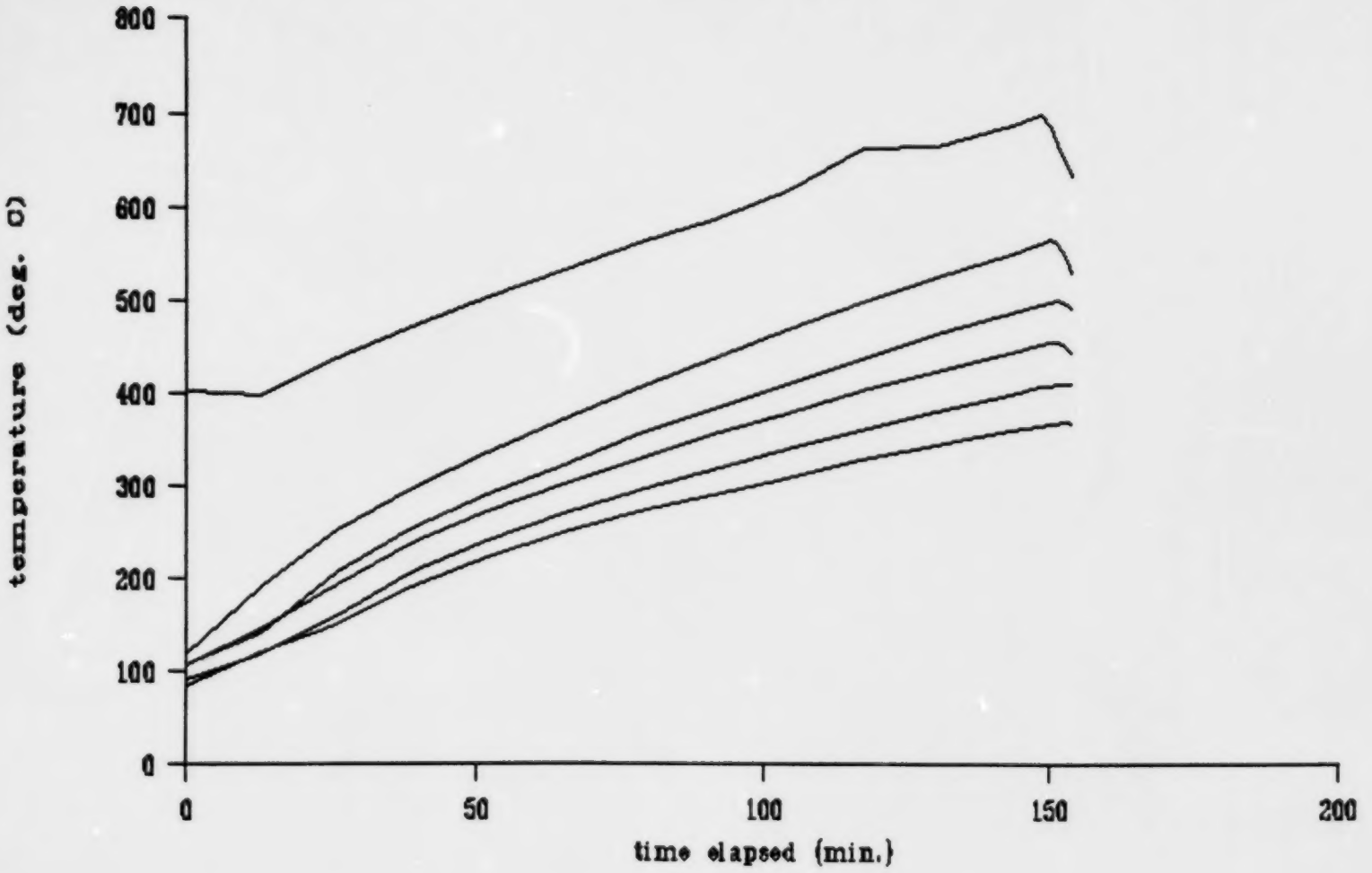


Figure 6 - Prince Mine PRC5 Coal

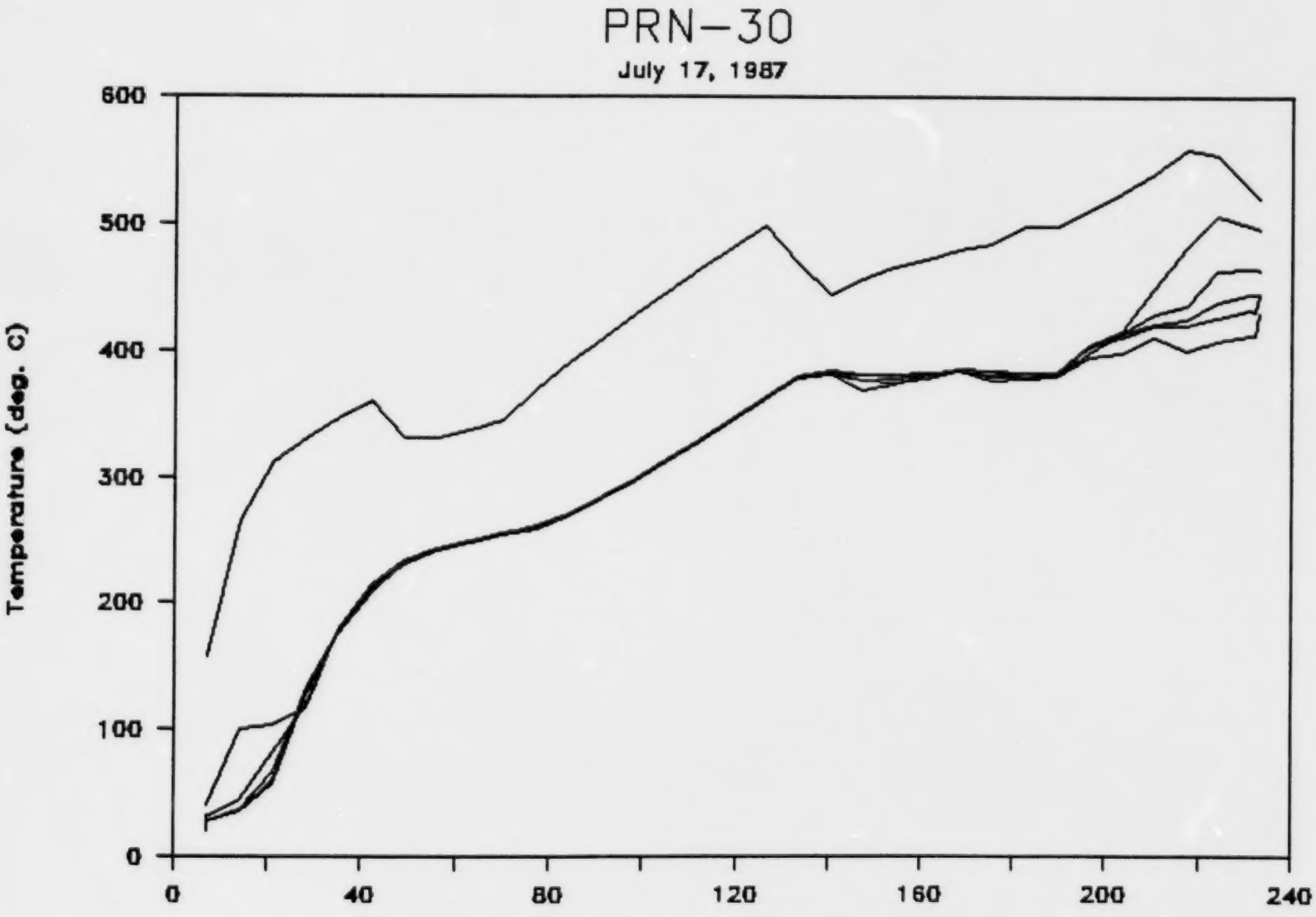


Figure 7 - Prince Mine PRN-30 Coal Slurry

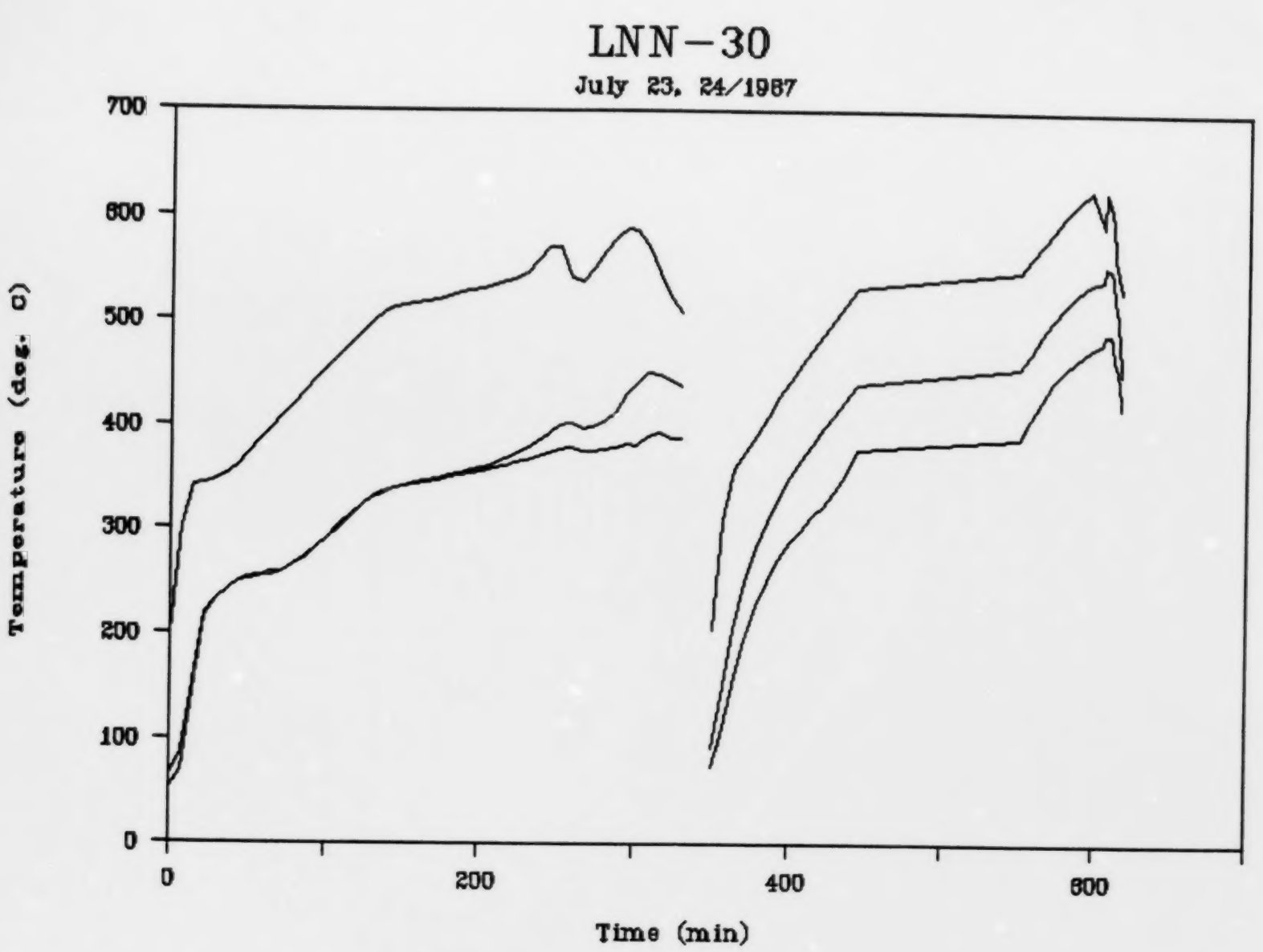


Figure 8 - Lingan Mine LNN-30B Coal Slurry

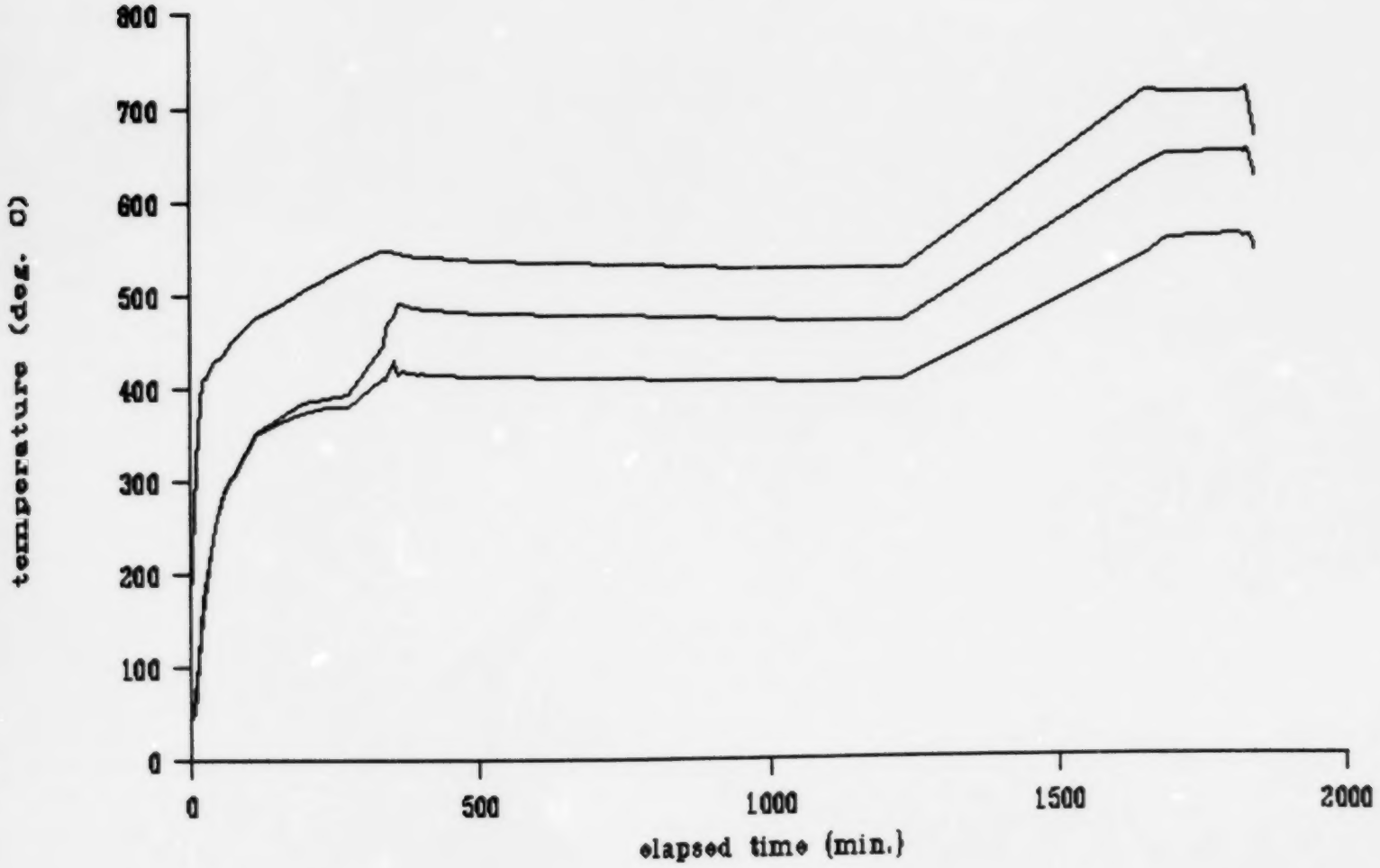


Figure 9 - Lingan Mine LNN-30C Coal Slurry

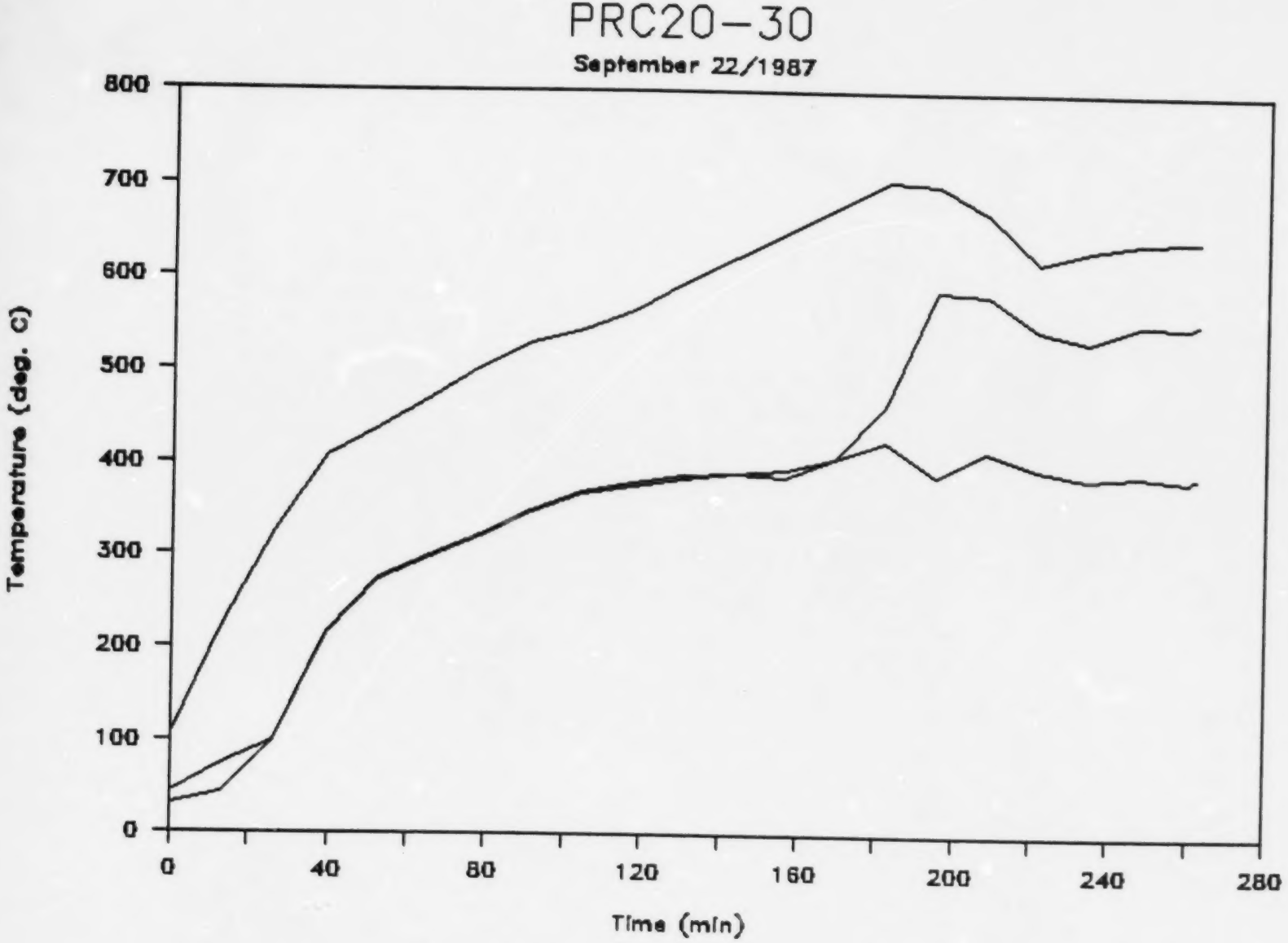


Figure 10 - Prince Mine PRC20-30 Coal Slurry

Microscopic Investigation

The results from the microscopic examination of the coal and slurry platofrost samples are summarized in Tables 3 through 6. The characteristics studied were grain fusion, pore development, optical texture, and the distribution of the CANMET additive. The characterization of pore development was difficult to quantify because of the small size and irregular shape of the pores in the coal grains. These grain sizes were much smaller than those used in our other Plastofrost studies. However, some generalizations regarding this property will be made. Grain fusion was sometimes preceeded by visible softening or rounding of the individual grains and this occurrence was noted. Complete grain fusion was identified when the individual grain boundaries could no longer be distinguished. Of course, all of these microscopic observations are subjective. The optical texture of a sample was characterized by the presence, shape and size of anisotropic material. Grint, Swietlik and Marsh[4] presented a classification system describing the isotropic and anisotropic components of the optical texture found in cokes. This is given in Table 4.

Table 5 presents a summary of the results for coke samples made with Forestburg sub-bituminous coal. This coal, as expected, did not soften, fuse or develop anisotropic texture in either the coal samples or the slurry samples. Photos A and B in Figure 11 show virtually unchanged coal with and without the CANMET additive. Photo B illustrates the difficulty encountered in visually identifying the iron sulphate which probably appears as tiny bright white or yellowish specks. Identification is based on the observation that

TABLE 4: Classification of the Optical Texture in Cokes [4]

Basic Anisotropy (b)	**a flat featureless anisotrope associated with the parent uncarbonized vitrain from anthracites. This changes little (apparently) during carbonization.**

Isotropic (I)

Fine-grained mosaics (Mf) < 1.2 microns in size

Medium-grained mosaics (Mm) < 5 microns in size

Coarse-grained mosaics (Mc) > 5 microns in size

Coarse-flow anisotropy (CF) elongated units > 5 microns in size

Flow/Domain (FD) large elongated shaped units > 60 microns in size

Domains (D) large rectangular shaped unit > 60 microns in size
D_1 is from basic anisotropy of low-volatile caking vitrains
D_2 is by growth of mesophase from fluid pitch

TABLE 5: Results from the Microscopic Examination of Forestburg Plastofrost Samples

Plastofrost Sample	wt % Additive in Coal	Initial Fusion or Softening	Completion of Fusion	Coal Optical Texture	Pitch Optical Texture	Additive Distribution	Comments
FBN	0	None	None	I			No softening or anisotropy developed Pores developed with increasing temperature but were small and irregular in shape. Reflectance increased with temperature.
FBC5	5	None	None	I		Not Visible	Same as above
FBC20	20	None	None	I		Very little Additive Observed Photos 2 & 4	No effect of the additive on this non-coking coal was observed.
FBN-30 540-586°C (25 min)	0	None	None	I	Mn		Unsoftened isotropic coal cemented in anisotropic pitch coke which had a medium grained mosaic texture.
FBC5-30 554°C (15 min)	5	None	None	I	Mn		Same as above
FBC20-30 569°C (80 min)	20	None	None	I	Mf		Coal grains were the same as above but the pitch coke had a fine grained mosaic texture, apparently as a result of the additive.

TABLE 6: Results from the Microscopic Examination of Illinois #6 Plastofrost Samples

Plastofrost Sample	wt % Additive in Coal	Initial Fusion or Softening	Completion of Fusion	Coal Optical Texture	Pitch Optical Texture	Additive Distribution	Comments
ILLN'A'	0	None	None	I			Coal did not soften, fuse or develop anisotropic texture but some small pores were observed at elevated temperatures.
ILLN'B'	0	None	None	I			Same as above
ILLC5	5	None	None	I		Not visible	Same as above
ILLC20	20	Some slight grain rounding	None	I		Additive is yellowish framboid structure - regions of high conc.	Same as above but some slight grain rounding may have occurred
ILLN-30 550°C for 50 min.	0	None	None	I	Mn		Unsoftened, isotropic coal cemented in a medium grained mosaic texture
ILLC5-30 'A' 17 hrs @ 460°C ½ hr @ 552°C	5	None	None	I	M_f-Mn	Additive barely visible as tiny bright specks	Same as above but pitch coke had more fine grained mosaic as well as medium grained mosaic
ILLC5-30 'B' 20 min @ 558°C	5	None	None	I	M_f-Mn		Same as above
ILLC5-30 'C' 7 hrs @ 520°C 10 min @ 650°C	5	grain rounding	None	I	M_f-Mn		Same as above but some grain rounding may have occurred
ILLC20-30 25 min @ 562°C	20	None	None	I	M_f-Mn		Same as above but grain rounding not observed

Table 7: Results from the Miscropic Examination of Prince Plastofrost Samples

Plastofrost Sample	wt% Additive in Coal	Initial Fusion or Softening	Completion of Fusion	Coal Optical Texture	Pitch Optical Texture	Additive Distribution	Comments
PRN	0	None	None	I			Pyrite framboids observed. Coal did not soften, or fuse, or develop anisotropy. Small pores developed at elevated temperatures.
PRC5	5	None	None	I		Additive not evenly distributed Areas of high conc.	Same as above
PRC20	20	None	None	I		Additive is more evenly distributed at grain boundaries	Same as above
PRN-30 'A' 15 min @ 408°C	0	None	None	I	I		Pitch not coked
PRN-30 'B' 30 min @ 506°C	0	None	None	I	M_f-Mn		Pyrite framboids unsoftened, isotropic coal cemented in pitch coke with a fine-medium grained mosaic.
PRC5-30 40 min @ 559°C	5	None	None	I	Mn	Not visible	Same as above but pitch coke is all medium grained mosaic due to the higher temperature.
PRC20-30 15 min @ 588°C	20	Softened	Partly Fused	I	Mn	Difficult to distinguish additive from inherent inerts	Coal had softened and partly fused probably due to higher temperature than above slurries. This may indicate that above coal samples were oxidized or that CLVB contributed to coal softening.

TABLE 8: Results From the Microscopic Examination of Lingan Plastofrost Samples

Plastofrost Sample	wt% Additive in Coal	Initial Fusion or Softening	Completion of Fusion	Coal Optical Texture	Pitch Optical Texture	Additive Distribution	Comments
LNN 'A'	0	1st fusion @ 370°C	380°C	M_f			Coal behaves like normal coking coal
LNN 'B'	0	1st fusion @ 365°C	386°C	I and M_f			Similar to above but less anisotropy, probably due to coal oxidation
LNC5	5	1st fusion @ 395°C	420°C 420°C	Mostly I very little M_f		Accumulates in voids, i.e. pores, cracks	Softening and fusion delayed and anisotropy reduced, probably due to additive
LNC20	20	1st fusion @ 409°C	partly fused	Mostly I very little M_f		Well dispersed at grain boundaries	Temperature data estimated due to failed data disk. Same as above.
LNN-30 'A' 17 hrs @ 350°C 30 mins @ 460°C	0	grain rounding	very little fusion	I	M_f		Coal was isotropic, probably due to low temperature
LNN-30 'B' 45 min @ 440°C 15 min @ 554°C	0	softened	partly fused	Mostly I at bottom	Mn		Coal and pitch anisotropy increased due to higher temperature
LNN-30 'C' 10 hrs @ 480°C	0	softened	mostly fused	mainly I at Mf above 5mm	Mn-Mc		Fusion increased and pitch coke anisotropy increased due again to higher temperature.
LNC5-30 540-567°C 2 hours	5	some softening	partly fused	I at bottom some M_f above	Mn-Mc	Not visible	Same as above but fusion reduced probably due to additive
LNC20-30 30 min @ 552°C	20	mostly softened	partly fused	I at bottom some M_f	Mn-Mc		Same as above

Figure 11: Photomicrographs showing the effect of temperature and ferrous sulphate additive in plastofrost samples of Forestburg sub-bituminous coal (-75 micron). (magnification 650°C)

Photo A: Untreated Forestburg coal at 398°C

Photo B: Forestburg coal impregnated with 20 wt% ferrous sulphate t 350°C

Photo C: Untreated Forestburg coal at 561°C

Photo D: Forestburg coal impregnated with 20 wt% ferrous sulphate at 555°C

FIGURE 11: FBN and FBC20 Samples

A

B

C

D

some mineral matter including iron (sulphides/sulphates) imbedded in the maceral matrix of coal appears as intense white (or yellowish) spots or regions during petrographic examinations of coal. This was tested in our study by comparing polished samples of impregnated coal with samples of coal to which iron sulfate had not been added. Careful inspection of the photographs in Figure 11 (photos A to D) shows irregular patterns of tiny bright spots in photos B and D which are not as apparent in photos A and C, although some still appear. Unfortunately, it is not possible to distinguish the iron sulphate from the natural pyrite or micrinite in the coal. Thus, tiny bright spots and even larger areas can be seen in both the FBN and FBC20 photos.

Photos C and D show the small irregularly shaped pores which are largely absent in photos A and B. It was observed that these increased with temperature, that is, more were seen towards the bottom of the sample.

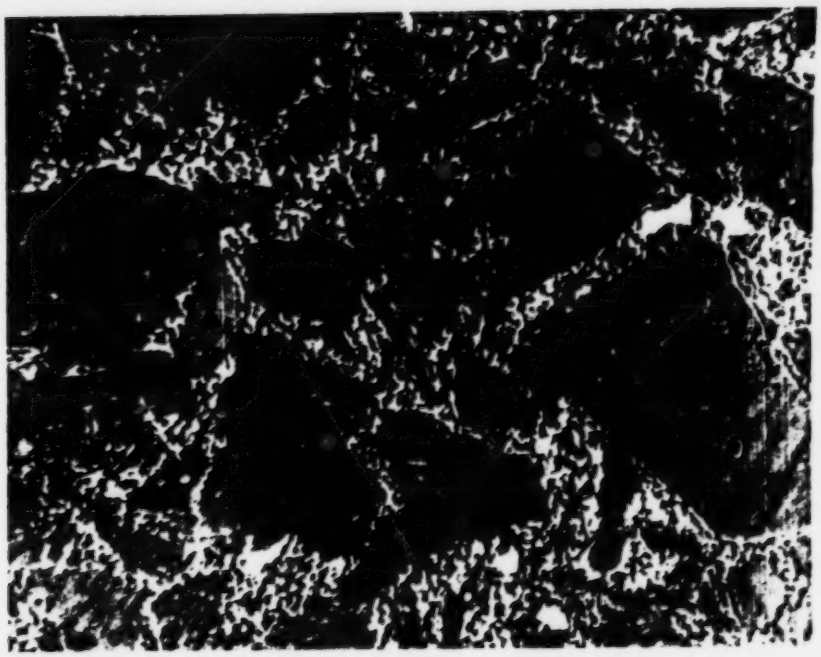
The sequence of photos A-C and B-D shows that the vitrinite regions (evident as the large light regions in the figure) become brighter as carbonization proceeds. This is frequently observed in petrographic studies of coal and semi-cokes.

The coked slurry samples yielded unsoftened, isotropic coal grains cemented in anisotropic coke from the pitch fraction. The optical texture of this pitch coke primarily consisted of medium-grained mosaics for FBN-30 and FBC5-30 and fine-grained mosaics for FBC20-30 which contained the most iron sulphate. This may be seen in Figure 12. Our observations suggest that the iron sulphate affects the formation of the texture of the pitch coke. There may be an effect on the temperature at which the anisotropic texture of the

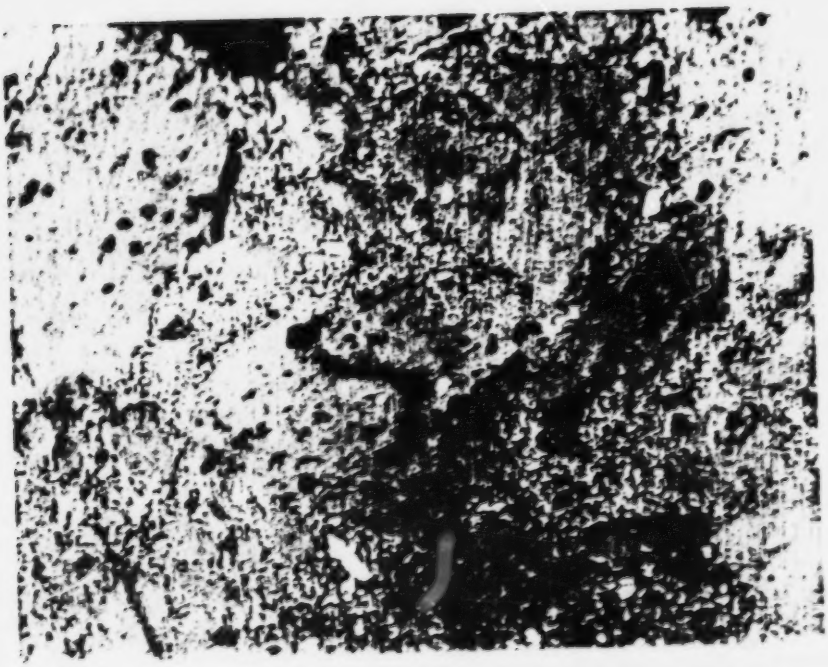
Figure 12: Photomicrographs of coked Forestburg slurry samples with and without ferrous sulphate (magnification 850x)

Photo A: Untreated Forestburg coal particles, unsoftened and isotropic, in a medium grained mosaic, anisotropic pitch coke (FBN-30)

Photo B: Unsoftened, isotropic Forestburg coal particles in a fine grained mosaic, anisotropic coal melt (FBC20-30)



A - FBN-30



B - FBC20-30

Figure 12: Forestburg Slurry Samples

pitch develops, but this could not be ascertained in the experiments performed.

Semi-coke formed from the Cold Lake vacuum bottoms is examined in Figure 13. All of these photos are magnified 650x. Photos A, B and D show different regions of a sample run at 554°C as discussed in the previous section. Photo C shows a region of a sample run at a lower temperature, 468°C.

Three types of mosaic structure can be seen. Photo A shows striking flow domain (mosaic) as defined by Grint et al[4]. Some regions of very coarse grained mosaic appears at the top and bottom of the photograph. A coarse grain and coarse flow mosaic appears in Photo D. This mosaic characterized most of the sample. Photo B illustrates the porous nature of the semi-coke which occupied all but the bottom 3 mm of the sample. A coarse flow anisotropy occurs around and between the pores although it is difficult to discern this mosaic in the photo.

Generally, there were few pores in the lower section of the sample. Pore number increased sharply by the mid-point of the section and continued to be numerous in the upper part. A boundary between lower pore numbers and high numbers seemed to be present. The explanation of this proposity variation is not known. It is surprising because the CLVB and slurry samples were at a roughly uniform temperature during these runs indicating that they were well mixed.

Photo C shows flow domain anisotropy for the sample coked at 468°C. Coalescing spheres in the upper part of the photo may be "liquid crystals"

Figure 13: Photomicrographs showing the structure and optical texture of coke from Cold Lake vacuum bottoms (magnification 650x)

Photo A: Flow domain anisotropy and some coarse grained mosaic in a CLVB sample coked to 554°C

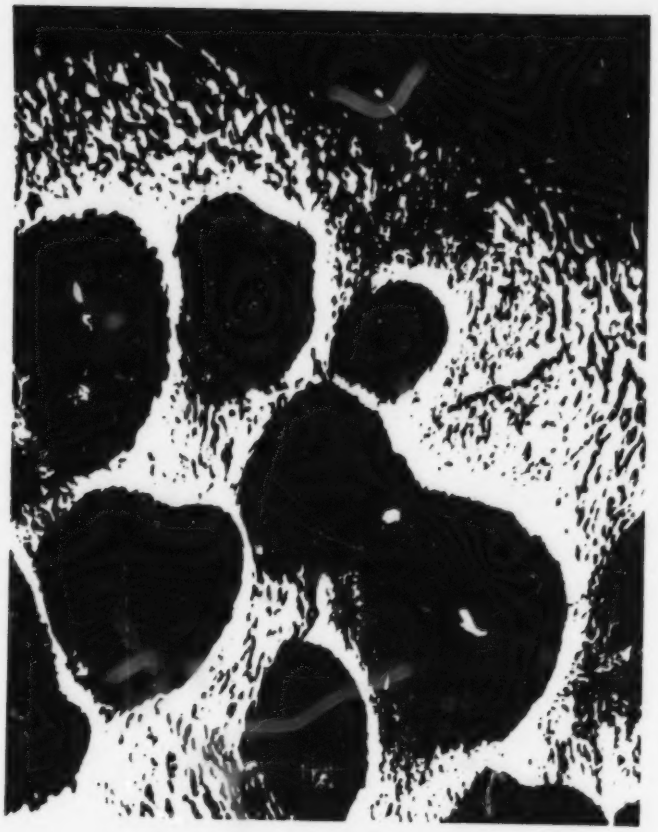
Photo B: Porous structure of the same sample with coarse flow mosaic

Photo C: Flow domain anisotropy and some liquid crystal (mesophase) coalescing in a sample of CLVB coked to 468°C

Photo D Coarse grained and course flow mosaic in the CLVB sample coked to 554°C

A

B



C

D

discussed by Marsh and co-workers[5].

The Prince Mine coal sample investigated was non caking. This is well illustrated in Figure 14. Photo A shows finely ground unaltered coal at the top end of the sample. The uniform grey region is vitrinite, bands on the right-hand side could be exinite, while the bright regions are either pyritic materials or inertinite macerals. The vitrinite becomes brighter in Photo B, taken at a temperature of approximately 441°C, but there is no smoothing of particle edges that signal softening. Photo C shows a cross section at a lower magnification (250x) at 541°C, near the bottom of the sample. The particle edges show no smoothing. Small pores can be seen in some of the particles. The upper middle region is enlarged in Photo D (magnification of Photos A, B, D, 650x). A comparison with Photo A shows some fine pore formation, but certainly no fusion. The lines are scratches originating during polishing.

Figure 15 shows photomicrographs of a region in Lingan Mine Plastofrost samples. The first three are at a magnification of 650x. The Lingan coal is caking, thus it is not surprising that particle fusion is clearly shown. Photo A is of the upper portion of the sample at a temperature of 367°C and shows fine coal grains at initial softening. Grain rounding and partial fusion is observed in this photo. The grey regions are vitrinite; some are banded with another maceral, probably exinite. The fine bright points are probably mineral or micrinite particles. Photo B, taken at 512°C shows complete fusion of the individual grains and some pore development. A fine mosaic seems to be developing. Note also that the individual grains have disappeared. Regions of a fine grain mosaic can be seen in Photo D (magnification x). It is evident that this fine mosaic is just barely distinguishable even at this high

Figure 14: Photomicrographs of Untreated Prince Mine Coal (PRN)

Photo A: Unaltered Prince coal showing vitrinite, exinite and inertinite heated to 350°C (650x)

Photo B: Unsoftened Prince coal with increased reflectance on heating to 441°C (650x)

Photo C: Unsoftened Prince coal coked to 541°C (250x)

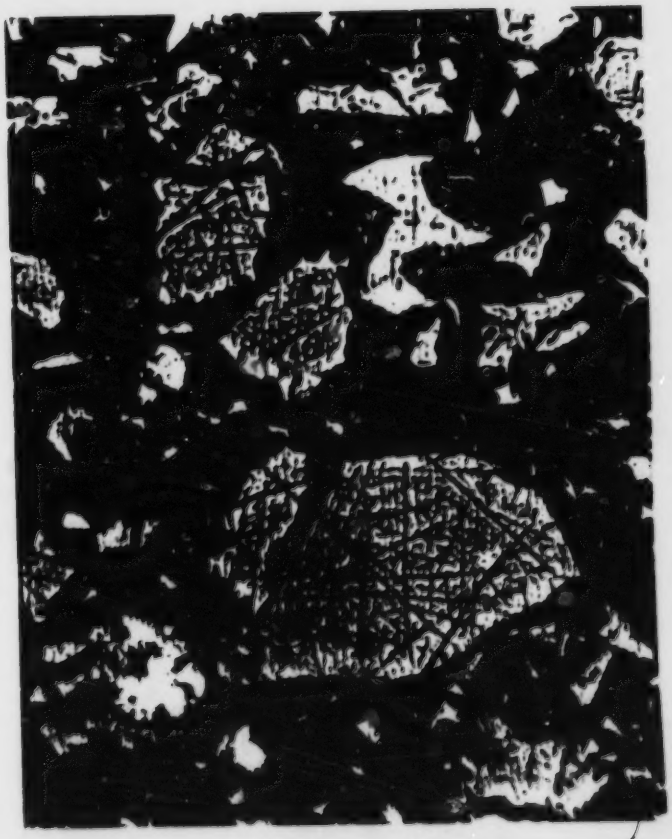
Photo D: Same as Photo C at 650x magnification

A

B

C

D



magnification and this is an indication of the high viscosity of the plastic phase of this coal.

Referring to Table 6 we find that both Lingan plastofrost samples (LNNA and LNNB) initially softened and fused between 365 and 370°C. Subsequent fusion was completed so that by 380-386°C individual grain boundaries could no longer be distinguished. However, the optical texture of the semi-coke formed was not exactly the same for both samples. It was observed that LNNA yielded a semi-coke consisting of approximately 90% fine grained mosaic and 10% isotropic material whereas sample LNNB was observed to have a semi-coke containing less (~60-70% Mf) fine grained mosaic and more isotropic material. The probable explanation lies in the fact that LNNB was coked about one month after LNNA was tested. It is probable that even with storage under nitrogen in a freezer the Lingan sample underwent enough oxidation to diminish its capability to produce anisotropic coke. This phenomena is probably general to all of the coal samples and causes the run date to become an important factor in the study.

The effect of the ferrous sulphate additive is examined in Figure 16 for Lingan Mine coal. Lingan coal prior to extensive fusion is shown in photos A and C of Figure 16. Photo A taken at 395°C shows the initial fusion of the Lingan sample with 5 wt% additive as bridges between the grains can be seen. For the 20 wt% additive sample no softening or bridging is apparent, however, the temperature at the point the photomicrograph shows is about 360°C. Photos B and D show the appearance at the bottom of the samples with temperatures at about 520°C. The figures are typical and they show a striking difference attributable, it seems, to the additive. Photo A shows that with 5

Figure 15: Photomicrographs Showing the Transition from Coal-to-Coke of an Untreated Lingan Mine Sample

Photo A: Lingan sample at 367°C showing initial softening of the vitrinite at 650x

Photo B: Lingan sample at 512°C showing completely fused coal mass with a fine grained mosaic texture at 650x

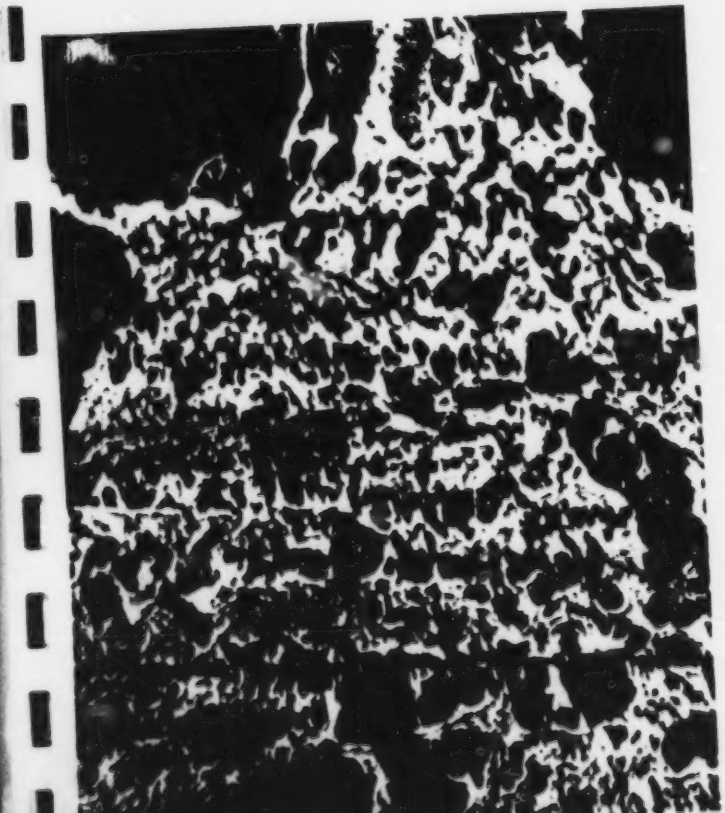
Photo C: Lingan sample at 580°C showing developed pores of semicoke at 650x

Photo D: Lingan sample showing the fine grained mosaic at a magnification of 1600x

A

B

C



D

Figure 16: Photomicrographs of Lingan Coal Treated With Ferrous Sulphate Additive (650x)

Photo A: Lingan sample with 5 wt% ferrous sulphate additive at 395°C showing initial softening and fusion (LNC5)

Photo B: Lingan sample with 5 wt% ferrous sulphate additive at 516°C showing mostly fused semicoke with additive (LNC5)

Photo C: Lingan sample with 20 wt% ferrous sulphate at approximately 360°C (LNC20)

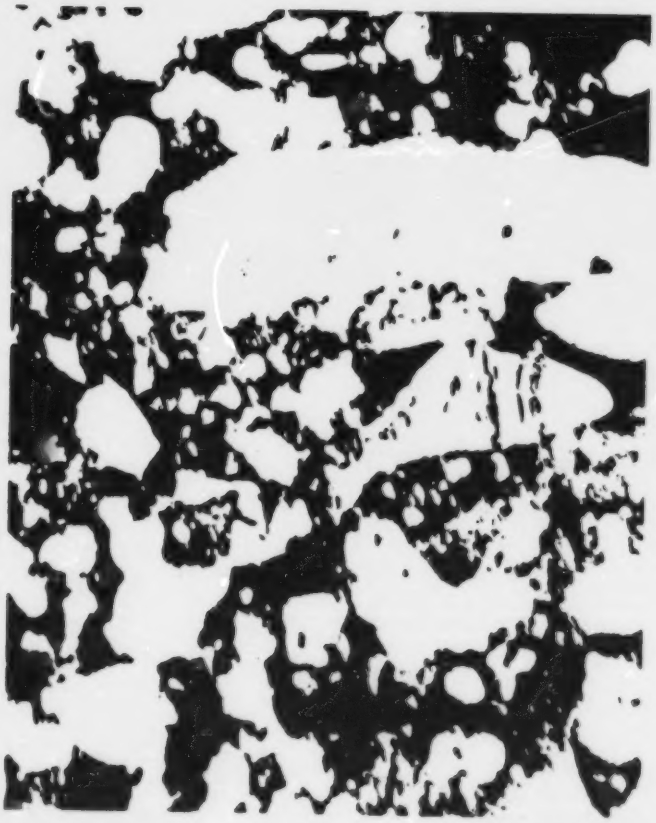
Photo D: Lingan sample with 20 wt% ferrous sulphate at about 550°C showing softened but only partly fused semicoke (LNC20)

A

B

C

D



wt% additive extensive particle fusion has occurred, but larger aggregates of fused particles can still be seen. With 20 wt% ferrous sulphate, particle softening and some bridging is apparent. Most of the particles, however, are separate, although jagged edges, seen in Photo C, are gone. The particles have evidently softened. The contrast of Photos B and D in Figure 16 with Photos B and C in Figure 15 with no additive, is remarkable. With zero ferrous sulphate, particles fuse completely and all individual identity is lsot.

It has also been observed that the presence of 5 wt% ferrous sulphate in the Lingan coal delays initial softening until 395°C compared to 365-370°C for untreated Lingan coal. The completion of fusion was also delayed until 420°C compared to 380-386°C. The nature of the fused mass also differs as illustrated by a comparison of photo B in Figure 16 with photo B or C in Figure 15. Similar observations can be made for the Lingan sample treated with 20 wt% ferrous sulphate. Initial softening was delayed until 409°C and the softened coal never completely fused. The optical texture of the semi-cokes formed from the impregnated samples contain less fine grained mosaic and more isotropic material than the untreated samples.

These observations suggest that the ferrous sulphate interferes with particle softening, fusion, and the subsequent development of anisotropic material.

Additive, apparently, is not visible under the microscope at 650x. A comparison of photo A in Figure 15 with photos A and C in Figure 16 show essentially identical appearance, even though they represent samples with 0 5 and 20 wt% additive. Ferrous sulphate must be very uniformly distributed on

a micron scale over the grain surfaces to be invisible at 650x. The occasional bright spots in the photos probably represents natural inorganic matter or inertinite macerals in the coal. Photos B and D in Figure 16 show bright dots distributed mainly around the boundaries of the softened and partially fused particles. These bright spots are largely absent in photos B and C in Figure 15 for which additive was not added. These spots may be additive.

Our explanation for the above observations is that ferrous sulphate is absorbed uniformly on the coal grains and possibly into the fine pore structure of the grains. It is invisible under the optical microscope for these reasons. Upon softening and melting, we believe the sulphate is forced out of the pore. This, together with agglomeration forced by reforming of the particle surface as it softens yields the fine additive particles seen in photos B and D of Figure 16.

This can be only a tentative explanation. An attempt was made to examine iron distribution in the samples using an X-ray analyzer equipped Scanning Electron Microscope. In principle, iron would appear as a matrix of fine dots on the sample surface if it is well distributed. Regions of the Plastofrost samples were found showing well distributed iron, but nearby regions did not exhibit this feature. The reason for the inconsistent results seems to be in sample preparation. The only diamond saw available for cutting samples was water cooled and the polishing equipment available also used water so leaching of the ferrous sulphate certainly occurred. Our attempt to use the SEM and an X-ray analyzer to investigate additive distribution was not successful. We believe the technique has promise, however. Much more effort will be needed to develop the procedure than we were able to expend.

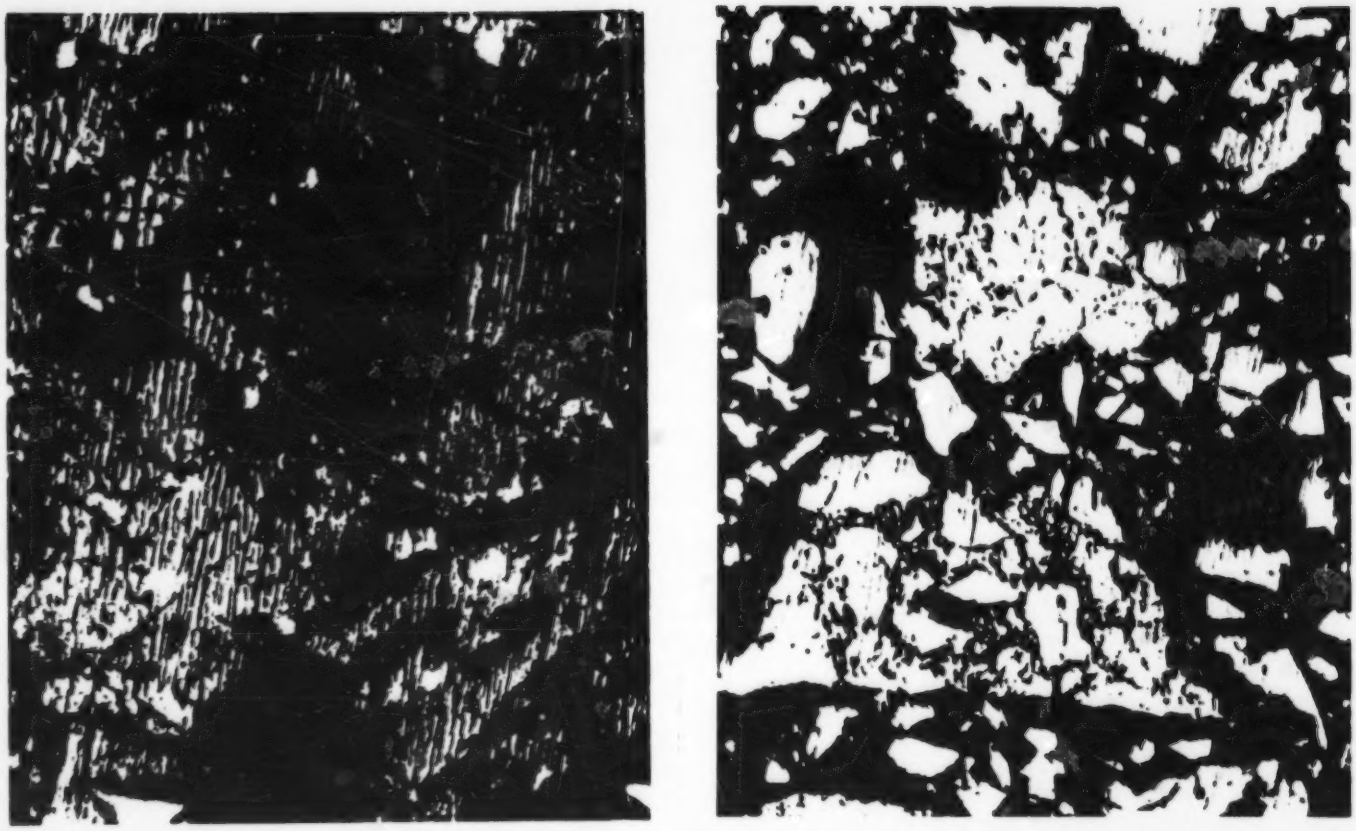
This work was not part of our subcontract with Cyclone Engineering.

An alternate, but less plausible explanation of the bright points in photos B and D of Figure 16 is that they represent abrasives used for sample polishing. Plastofrost samples from additive impregnated coal were cleaned with alcohol, whereas samples from raw coal were cleaned in water. Alcohol caused some softening of the epoxy resin and perhaps this trapped small amounts of the carbide or alumina particles.

Figure 17 shows photomicrographs taken of Prince Mine coal with 20 wt% ferrous sulphate at the top of the sample (T = 379°C) and at the bottom (T = 565°C). Apart from the brightening of the vitrinite macerals, there seems to be no change. Indeed, these figures are comparable to photos A and D in Figure 14 taken of the Prince Mine coal without additive. The additive appears to have no effect on the carbonization process. Although figures for the Illinois coal have not been included, they too show no change in the carbonization process due to the presence of ferrous sulphate.

In the next sequence of photomicrographs, we examine the plastofrost results with slurries of CLVB Prince Mine Coals. Figure 18 illustrates some of the observations made with these slurries. Photo A shows the typical appearance for sample PRN-30A for which heating was halted at 408°C. Boiling off of the vacuum bottoms has probably not finished. The pitch has not carbonized as evidenced by the absence of any mosaic texture. The roughened appearance results from polishing the relatively soft material. Unsoftened, isotropic coal (vitrinite) particles are clearly evident. Results for the same slurry coked to 506°C, however, show that the pitch has carbonized

Figure 17: Photomicrographs of Prince Mine Coal with 20 wt% Ferrous Sulphate Additive (650x)



A B

Photo A: Photomicrograph of PRC 20 sample at 379°C

Photo B: Photomicrograph of PEC20 sample at 565°C showing unsoftened but brighter isotropic coal

to give a fine to medium grain anisotropic coke seen in photo B. The large black region is a pore. Pores, typically, are surrounded completely by anisotropic coke. Coal particles, which are largely vitrinite, appear as the featureless isotropic regions. Coal grains tended to settle out and concentrate in the bottom of the slurry. Smaller pores, usually in the neighbourhood of coked pitch can be seen as well as inorganic materials (mainly pyrite) or inertinite macerals. These are evident in both photos A and B.

Photo C also shows the bottom region of a Plastofrost slurry sample, however, this slurry contained Prince coal treated with 20 wt% ferrous sulphate and the final Plastofrost temperature was 588°C rather than 506°C. The high concentration of coal in photo C compared to photo B illustrates the settling of the coal in the slurry samples. This bottom region is also relatively free of large pores. The higher temperature, and the higher coal concentration, has resulted in softening and partial fusion of the coal grains, however, they remain isotropic. The fine-medium grained pitch coke seems to inhibit particle fusion particularly in upper regions of the sample (with reference to the heating plate) where the coal is more dilute. There is no evidence suggesting an additive effect. The observation of coal softening contrasts with all the other Prince tests in which the coal did not soften. This could be due to a combination of factors including high temperature, unoxidized coal, slurry and/or additive effects.

Our examination of the other non-caking coals, Illinois and Forestburg, lead to observations largely the same as those discussed above. Photo D in Figure 18 shows a Illinois slurry sample coked at 650°C. Individual isotropic coal particles are seen in a fine grained mosaic pitch coke just as with the

Figure 18: Photomicrographs of Prince and Illinois #6 Coal Slurried in CLVB (650x)

Photo A — Typical appearance of sample PRN-30A coked at 408°C showing unsoftened, isotropic coal particles cemented in isotropic CLVB

Photo B — Sample PRN-30B coked at 506°C showing isotropic coal particles in a CLVB semicoke with a fine-medium grained mosaic and a large pore

Photo C — Sample PRC20-30 coked at 588°C showing partly fused isotropic coal particles in a medium-grained mosaic pitch semicoke

Photo D — Sample ILLC5-30C coked at 650°C showing unsoftened, isotropic coal particles in a fine-medium grained mosaic pitch semicoke

A

B

C

D

Prince samples.

The remaining figures in this section illustrate the appearance of slurries of Lingan Mine coal all at 30% by weight coal. Figure 19 shows regions from the top to the bottom of samples made with a coal slurried in vacuum bottoms which had not been treated with ferrous sulphate. The final temperature was about 554°C.

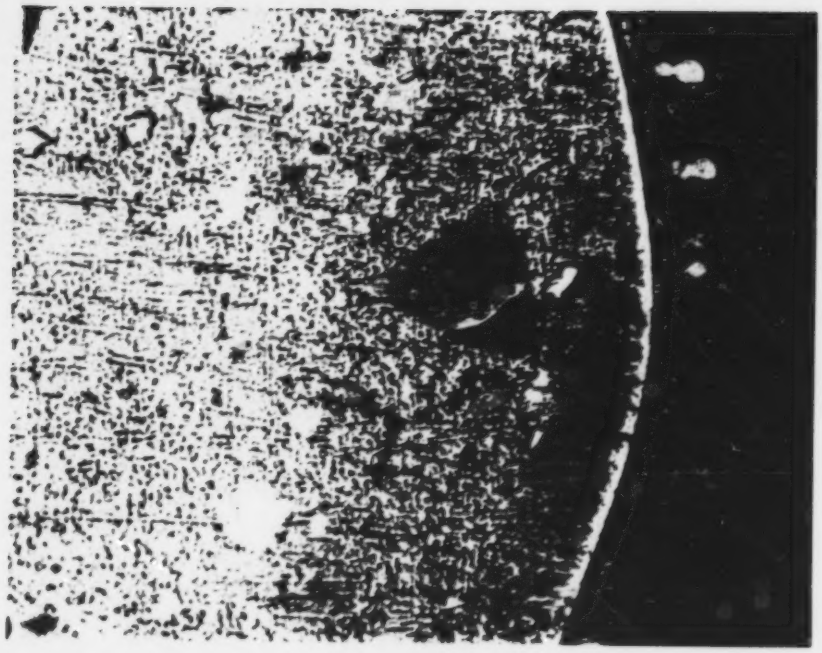
Beginning with photo A showing the top of the sample, the upper surface can be seen. There appears to be a barrier of isotropic material, uniform in texture and colour, at the surface. The dark region immediately below is probably a crack or peeling of this layer which occurred during cooling. The spheres must be condensate droplets that have fallen downward and become trapped on, but not absorbed into the top layer. The medium grained mosaic is semicoke formed from the vacuum bottoms. This can be seen better in Photos B and C. Some of the coal (vitrinite) particles are visible in photos A and B, but at the bottom of the sample softening and fusion have occurred as shown by photo C. Barely visible in photos A and B is a fine grain mosaic in some of the coal particles. The photos illustrate the progression from top to bottom in the sample and show the increasing coal concentration in the lower regions of the sample. No coal anisotropy was seen in the bottom region in contrast to observations higher in the sample. This may suggest a slurry effect on the development of coal anisotropy. Fusion of the coal particles at the bottom of the sample has taken place. Vacuum bottoms based coke surround the coal particles in Photos A and B may indicate that the bitumen phase is interfering with grain fusion. It may also be simply the result of the lower coal particle density and the looser packing in the upper part of the

Figure 19: Photomicrographs of Untreated Lingan coal (sample LNN-30B) Slurried in CLVB and Coked at 554°C (650x)

Photo A — Top surface of coked Lingan slurry sample showing some isotropic and some anisotropic (mf) coal particles in anisotropic (Mm) CLVB semicoke

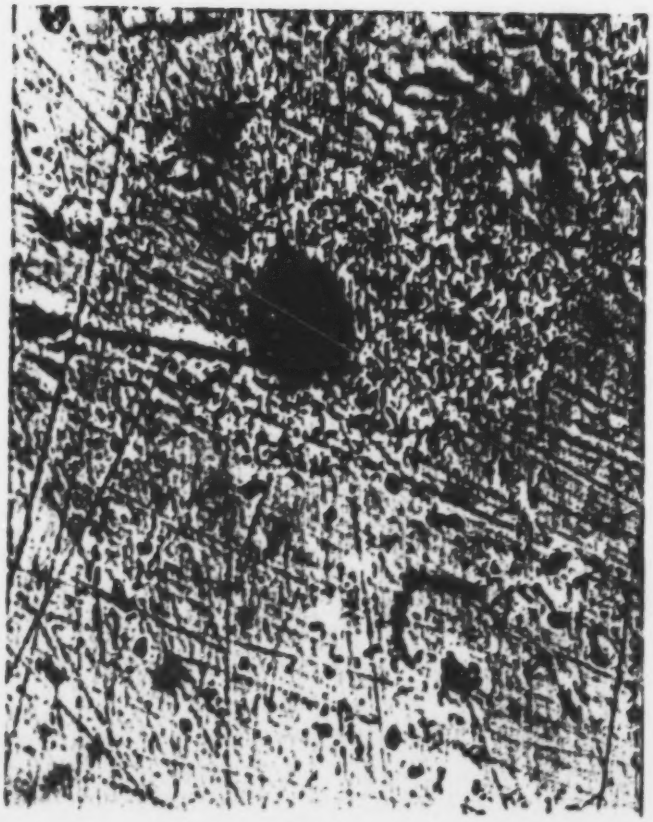
Photo B — Middle section of sample showing some softened coal (I & Mf) in CLVB semicoke

Photo C — Bottom of sample showing mainly fused isotropic coal with some anisotropic CLVB semicoke



A

B



C

sample. Inorganic material or inertinites are gathered in small regions as the photos show.

As summarized in Table 6 the Lingan slurry with no additive was coked at three different temperatures: 460°C, 554°C and 651°C. In comparing the samples from these runs it was observed that the scale of the anisotropic texture increased with increasing temperature. Whereas the CLVB semicoke in sample LNN-30A, coked at 460°C, displayed a fine grained mosaic, the LNN-30C sample coked at 651°C developed a coarse grained anisotropic texture. This is illustrated in Figure 20. In addition, the coal particles also developed some fine grained mosaic texture at 554°C and 650°C but remained isotropic at 460°C.

Photomicrographs of samples prepared from Plastofrost runs of slurries of Lingan Coal treated with 5 wt% ferrous sulphate are given in Figure 21. Photos A and B show regions of the bitumen upper part of the sample which exhibit a large numbers of pores. This is more typical of this portion of the coked slurry samples than Photo A in Figure 19. Pitch has formed a medium grained mosaic typical of an anisotropic coke. Photo A shows this coke surrounding circular pores, whereas coal particles border smaller pores as Photo B illustrates. Inspection of this photo shows that many of the vitrinite grains are separated by pitch coke, and there is ample evidence of softening and partial fusion of the coal in this picture (bottom part of the photo). A region of largely coarse grained mosaic pitch coke in the middle of the sample can be seen in photo C. Coal particles occur in the centre and cracks (on cooling) seem to be located in the region of these particles. Just like the coal particles in photo B, a fine grain mosaic can be seen in some of these

Figure 20: Photomicrographs Comparing Anisotropic Texture of Lingan Coal Slurries Coked at 460°C and 650°C

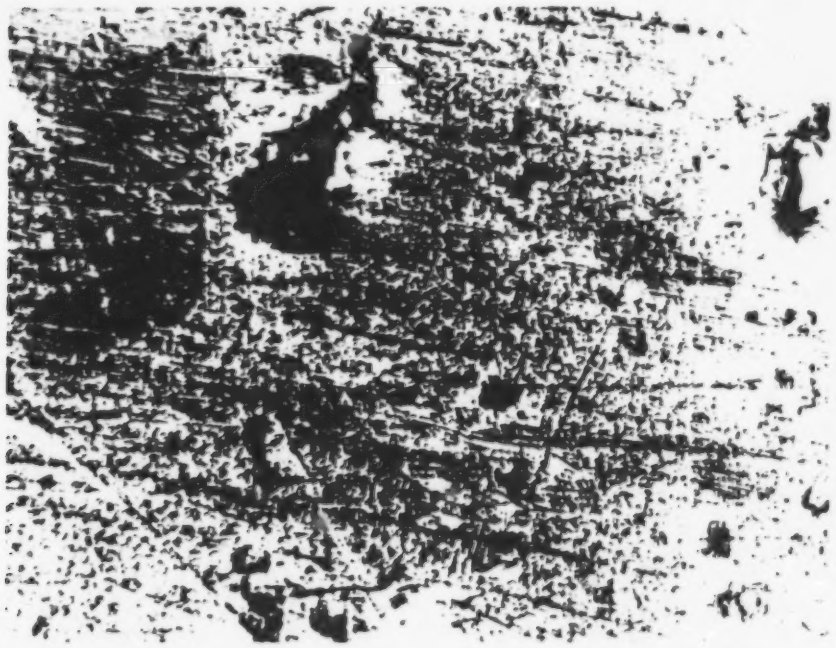


Photo A: Sample LNN-30A coked at 460°C showing unsoftened isotropic coal particles in a fine grained mosaic pitch semicoke

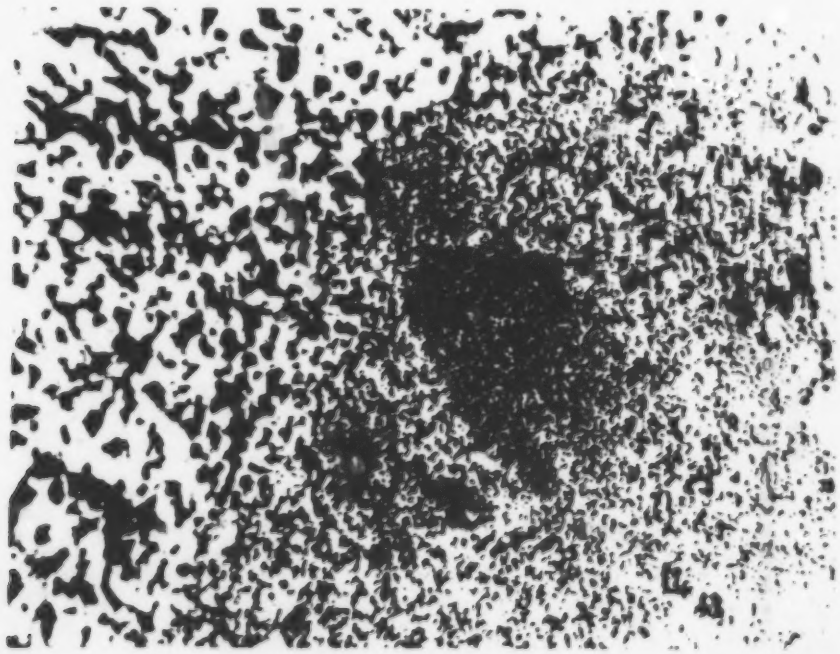


Photo B: Sample LNN-30C coked at 651°C showing isotropic and anisotropic coal particles in a fine-grained mosaic pitch semicoke

particles.

Photo D shows a region at the bottom of the sample with cooling cracks. Coal particles are closely packed in this region and very little pitch coke is visible. The pitch coke in some parts of the photo shows the boundary of coal particles; nevertheless most of the particles have fused into a single isotropic mass.

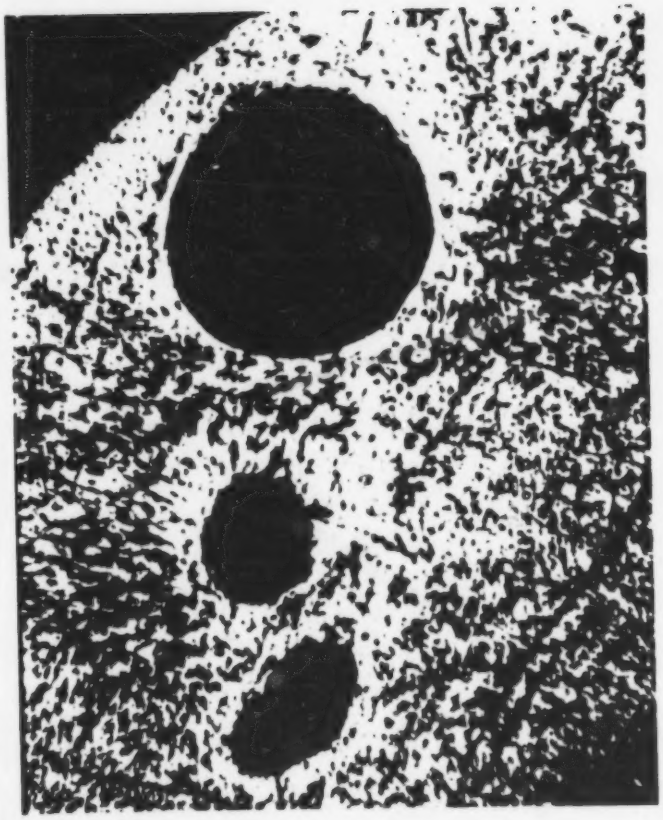
Photos of Plastofrost samples from Lingan coal treated with 20 wt% ferrous sulphate and slurried with CLVB are shown in Figure 22. The upper boundary of the sample may be seen in photo A. Condensate droplets and a surface layer are evident, although this layer is very much thinner than in Photo A of Figure 19. Pitch coke predominates at this surface and exhibits a medium-coarse grained mosaic. Coal particles visible in Photo B illustrate that while some particles remain isotropic (lower centre), others have developed a fine grained mosaic texture making it difficult to distinguish them from the pitch coke. Close examination reveals that some grains have partially fused, while others are separated by pitch coke.

Photos C and D show regions from the bottom of the sample. Spherical pores surrounded by fine-medium grained mosaic originating from the pitch is evident in Photo C. Densely packed isotropic coal is visible in the bottom of the photo and individual particles are separated for the most part by pitch coke. Photo D shows this at a higher magnification and also reveals the development of a fine grain mosaic for some particles; other vitrinite particles are featureless and isotropic.

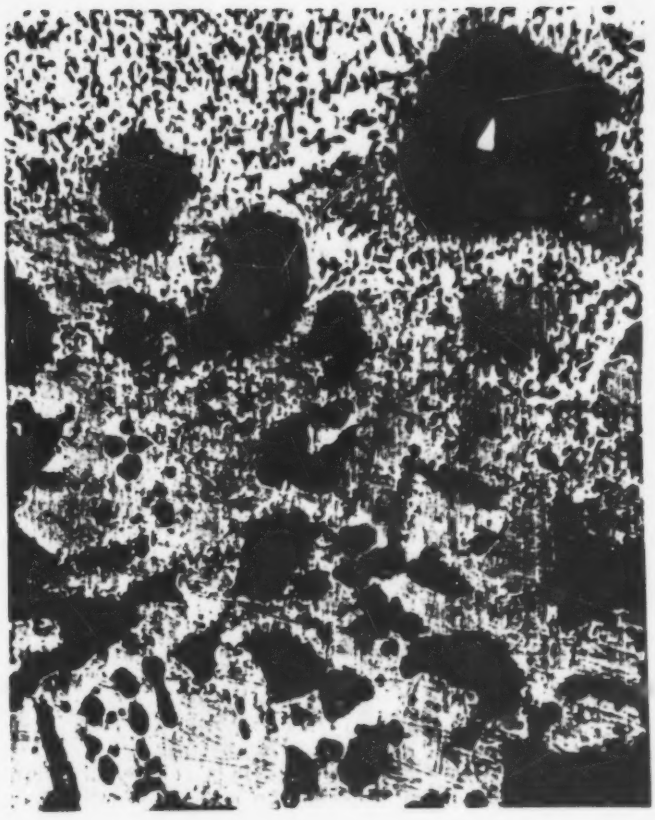
Figure 21: Photomicrographs of Lingan Coal Treated with 5 wt% Ferrous Sulphate, Slurried in CLVB and Caked to 567°C (650x)

Photo A	**Top surface of sample showing mainly CLVB semicoke with a medium grained mosaic texture and large pores**
Photo B	**Top of plate shows anisotropic semicoke (Mm) while middle and bottom regions consist of mostly isotropic, partly fused coal**
Photo C	**Coarse grained mosaic CLVB semicoke with some coal and semicoke particles in crack**
Photo D	**Bottom of sample showing mainly isotropic, fused coal with fissures**

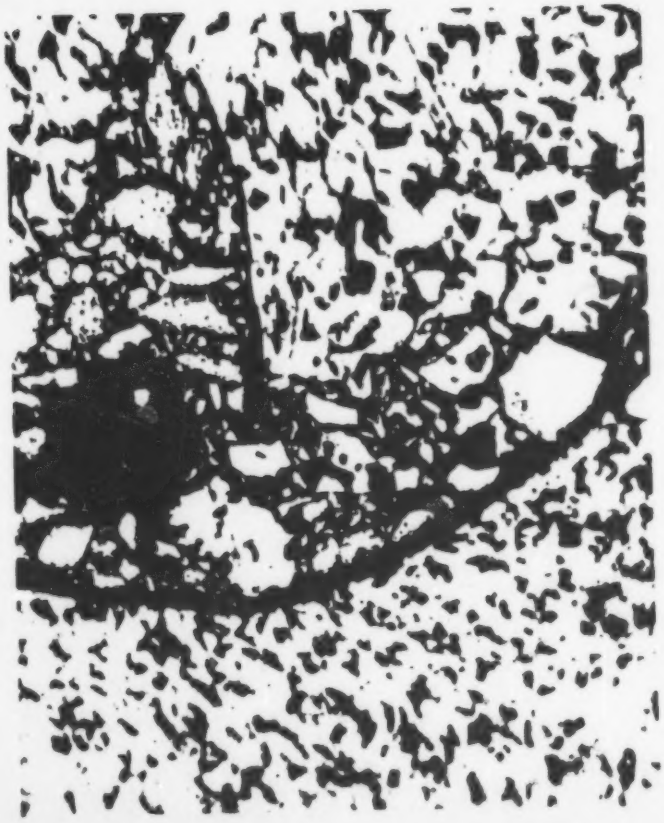
A



B



C



D

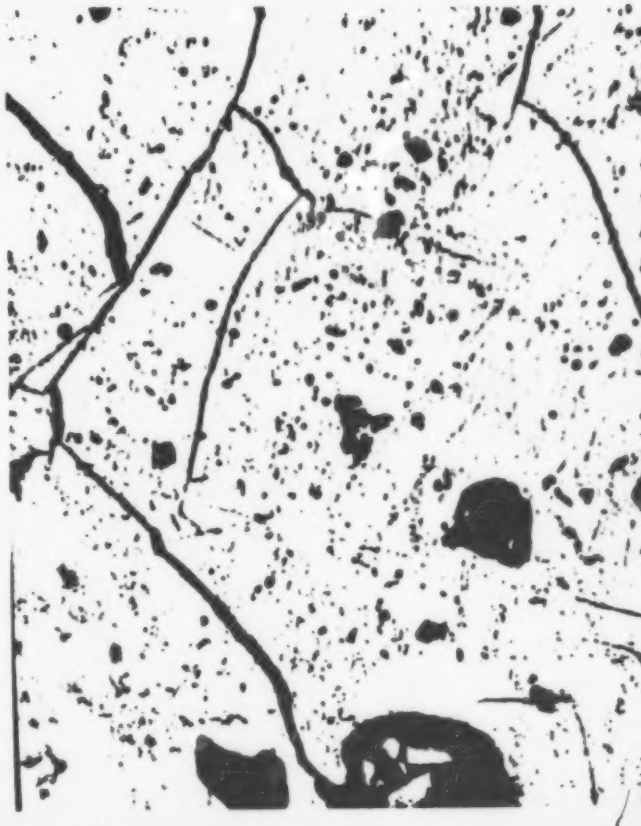


Figure 22: Photomicrographs of Lingan Coal Treated with 20 wt% Ferrous Sulphate, Slurried in CLVB and Coked at 552°C

Photo A — Upper boundary of sample LNC20-30 with predominantly medium-coarse grained mosaic pitch semicoke

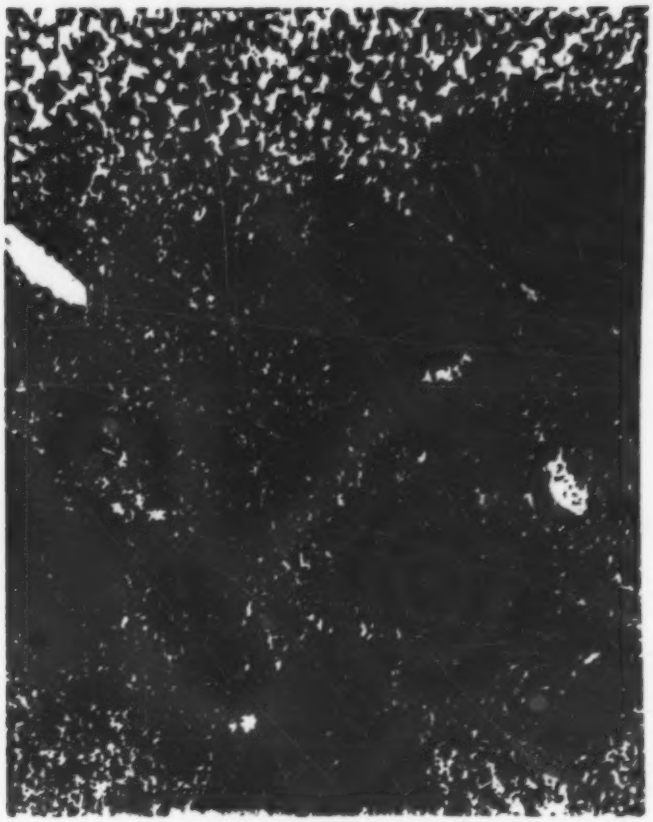
Photo B — Isotropic and fine-grained anisotropic coal particles in a medium-grained mosaic pitch semicoke

Photo C — Lower region of sample showing pores in a medium-grained pitch semicoke and densely packed isotropic coal particles

Photo D — Photomicrograph of sample LNC20-30 magnified 1600x showing the very fine-grained mosaic texture of some coal particles and the coarser texture of the CLVB pitch semicoke as well as some isotropic coal particles (1600x)

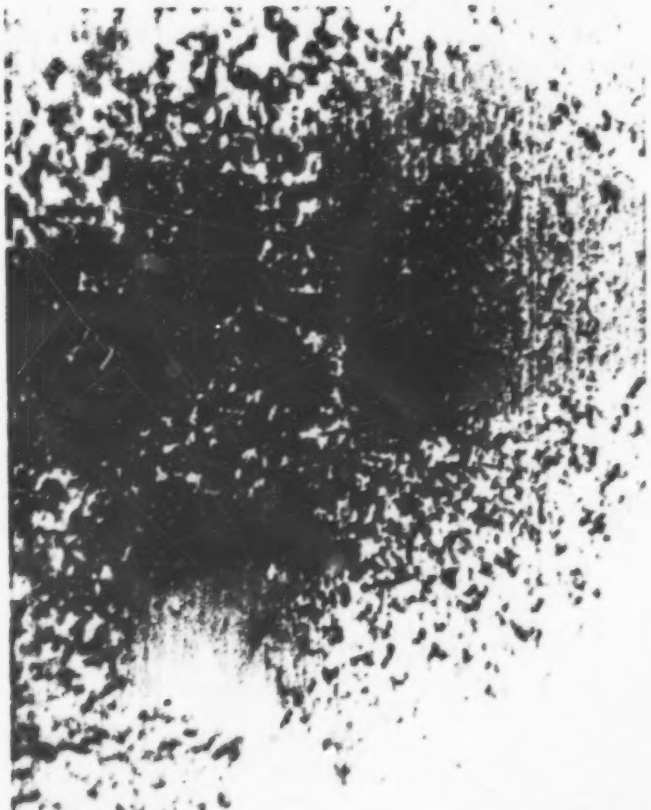
A

B



C

D



Interpretation of the Plastofrost study of the Lingan coal slurries with and without the iron additive leads to several conclusion without comparing the individual photomicrographs: there is no doubt that the pitch, after losing some 60% of its volume by boiling, forms a coke. There seems to be no solution of coal in the vacuum bottoms. Each phase retains its identity throughout the carbonization process. Pitch binds the coal particles and seems to form a coherent mass as cooling cracks infrequently appear along pitch coke and coal coke boundaries.

There may be a further effect. Plastofrost experiments with CLVB alone showed the coke formed exhibited either a coarse grain mosaic or flow anisotropy. These disappeared when the CLVB was slurried with coal. Indeed, with the three non-caking coals, a fine to medium mosaic occurred whether ferrous sulphate was present or not. Addition of additive to Lingan coal, however, led to regions of coarse grain anisotropy. Coarse grain mosaics also appeared in the slurry with untreated Lingan coked to 650°C. This did not occur with an Illinois coal slurry coked to the same temperature. It may be that the additive affects the oil-coal interaction in the coking process.

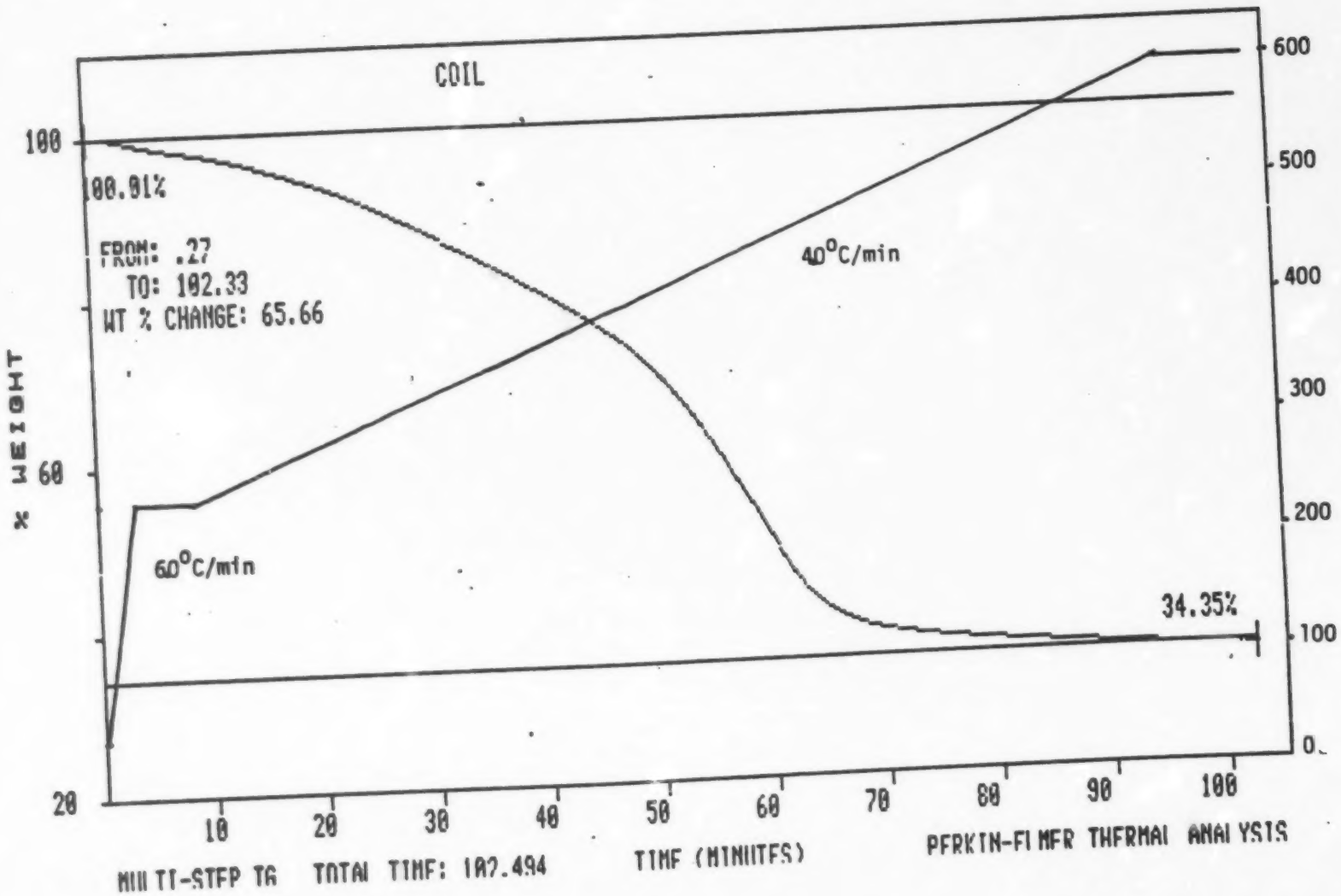


Figure 23: TGA Plot for LHOP

REFERENCES

1. Fouda, S.A. and Kelly, J.F., "Coprocessing of High Volatile Bituminous Coals", Proc. Int'l. Cont. on Coal Science, (ed. J.A. Moulign), Elsevier Science Publishers B.V., Amsterdam, 1987.

2. Ritter, H. and Juranek, G., "Eine Neue Methode zur Untersuchung und Beschreibung des Ereweichungsverhaltens von kohlen", Brennstoff chemie, 42, 17 (1961).

3. Duever, P.D., "An Investigation of Coal Rheology with a Plastofrost Apparatus", MASc Thesis, Department of Chem. Eng., University of Waterloo (1986).

4. Grint, A., Swietlik, U and Marsh, H., "Carbonization and liquid-crystal (mesophase) development.9. The co-carbonization of vitrains with Ashland A200 petroleum pitch? Fuel, v58, p.642 (1979).

5. Brooks, J.D. and Taylor, A.H., "The Formation of Some Graphitizing Carbons" in P.L. Walker, Jr. (editor) "Chemistry and Physics of Carbon" 4, 243-286 (Marcel Dekker, New York - 1968).

6. Marshall, C.E., Fuel, 24, 120 (1945).

APPENDIX A

EQUIPMENT AND PROCEDURE DEVELOPMENT

Several modifications to the original Plastofrost apparatus and operating procedures[3] were made prior to working with the coprocessing samples. The furnace was redesigned by replacing four strip heaters with high temperature cartridge heaters capable of operating at temperatures of nearly 1000°C. This allowed the copper slab to be reduced in size and eliminated the need for a steel support box. The overall effect, therefore, was to reduce heat losses and increase the heating capacity so that higher heating rates and higher operating temperatures could be achieved.

The coking attachment was also modified and a spare was built to reduce the time needed to prepare a sample. The effects of the modifications were to minimize heat losses and maximize heat transfer to the sample. Sample temperatures were monitored using chromel/alumel thermocouples protected by a 1/25" stainless steel sheath and this reduced the total run time by about one hour compared to the old method using bare thermocouple wire protected with ceramic tubes.

Some problems were encountered adopting the Plastofrost technique to the corprocessing samples. New methods were developed to produce Plastofrost samples suitable for microscopic analysis from the very fine (-75 micron) coprocessing coals. Coking the CLVB and coprocessing slurry samples required the development of completely new experimental procedures.

Following a standard coal Plastofrost run, the sample, held together with an aluminum foil liner, is carefully removed from the coking cylinder. It is then impregnated with a polyester resin by pouring the resin onto the top, uncoked portion of the sample and allowing the resin to percolate through the sample. Once the resin has hardened the sample is cut in half using a band saw and the exposed surface is prepared for microscopic analysis. However, because of the very small particle size of the coprocessing coals, the samples often fell apart when removed from the coking cylinder. Also, if the resin is poured onto the sample before it is removed from the cylinder the fine particle size prevents the resin from penetrating beyond the surface and the sample crumbles when removed from the cylinder and cut.

In response to these problems some modifications were made to the coal sample preparation procedure. First, the aluminum foil was replaced with a heavier gauge aluminum liner which provided greater support to the sample and allowed all samples to be removed from the coking cylinder without collapsing. Second, the resin was diluted with acetone to reduce its viscosity and allow it to percolate through the sample better. Also, very little hardener was added to the resin which gave it more time to penetrate the sample before it began to gel. Less coal (66.5g vs 72g) was packed into the cylinder before a run reducing the packing density and increasing the ability of the resin to percolate through the sample. Finally, a diamond-tipped circular saw was used to cut the samples instead of the band saw. This saw yielded a smoother surface and caused less disturbance of the sample than the band saw.

When processing CLVB and coprocessing slurries the coking cylinder must be sealed to prevent leaking of the hot bitumen during a run. This was

accomplished with a sodium silicate solution which when heated forms a brittle, glassy, solid and seals the seams of the cylinder. Also, if the bitumen is heated too fast it will boil rapidly and bubble over the top of the cylinder. The furnace heating rate was therefore controlled so that the bitumen boiling point was approached slowly. Another problem encountered during these Plastofrost runs was that between 60 and 70 percent of the bitumen was distilled off leaving a relatively small residue sample for analysis. Furthermore, the sample was well mixed so that the vertical temperature gradients in the coking cylinder were eliminated. Therefore, it was concluded that one or two thermocouples placed at the bottom of the sample would suffice to accurately monitor the sample temperature during a run. Since temperature gradients do not appear to be present in the sample, the size of the final coke product does not have to coincide with that from a coal run. Therefore, the standard 3" high coking cylinder was used for the CLVB and slurry tests, but the capacity was increased by extending the height of the sealed aluminum liner about 6 cm above the top of the cylinder. Also, with the standard ventilation of the plastofrost container the CLVB vapours reached fairly high concentrations within the container posing possible health and safety risks. To improve the ventilation, a 1" hose was inserted in the container and connected to the fume hood vent for suction. This vent, in addition to purging the box with nitrogen, kept the vapours at safe levels.

To determine the amount of solid residue which can be expected from a coprocessing coal-oil-slurry the PRN-30 slurry sample was coked in the thermogravimetric analysis instrument (TGA) under nitrogen at 4°C/min from 250°C to 600°C. The results from this test are shown in Figure 23. We can conclude that approximately one third of the slurry fed will remain as coke.

APPENDIX B

DESCRIPTION OF PLASTOFROST SAMPLES

COAL

FBN Fig. 11A (~298°C) no softening - possible oxidation rim (not typical)
Fig. 11C (~560°C) no softening - but some pore development

Pores developed steadily with temperature

Very difficult to determine initial pore development

No significant increase in pores

No anisotropy

FBC5 Same as FBN - no additive observed

FBC20 Fig. 11B(~350°C) unchanged coal possibly with bright specks-additive
Fig. 11D (~550°C) higher reflectance, more pores, no softening, additive? No anisotropy.

ILLNA Poor polish - no softening - pore development very difficult to characterize

No anisotropy

ILLNB No anisotropy - possibly some grain rounding?

Very little pore development evident

ILLC5 Same observations as ILLN

ILLC20 Some rounding of grains? (especially larger ones)

No fusion or pore development evident

No anisotropy

Regions of high additive concentration noticed

Additive appears as yellowish, framboid-like, darker than pyrite

PRN No softening (see photo #10-11)

Not much change with temperature observed

Fig. 14B @ 441°C no softening, no pores

Rims around grains - poor polish or oxidized?

Fig. 14A @ 353°C - unchanged coal: vitrinite, exinite, inertinite

Vitrinite reflectance increases with temperatures

Fig. 14C,D ~541°C - no softening, no anisotropy

Lots of pyrite - framboids

PRC5 No softening - no significant pore development

No anisotropy
Additive present in clusters of high concentrations
- not evenly distributed

PRC20 Fig. 17A @ 379°C No softening

Fig. 17B @ 565°C pore development but no softening

No significant pore development - no anisotropy

Additive more evenly distributed at grain boundaries

LNNA Visual porous - non-porous boundary at 381°C

Initial softening at x = 1.3 (370°C)

Complete fusion at x = 3.7 (380°C) - no grain boundaries

Fusion progresses from first fused particles to all grains bridged to other grains then disappearance of grain boundaries

Almost all semi-coke Mf ~85-90% - remainder is I

LNNB Some softening by 410°C

Additive seems to accumulate in pores or cracks of coke

Initial softening 394.7°C - just barely (small particle fused) larger particles softened by 414.2°C

Completely fused at 433°C

Pore development not evident

Predominantly isotropic - some Mf

LNC20 Initial softening by 409°C - very little pore development

Never completely fuses

Additive appears well dispersed on surface of coal

Predominantly isotropic, very little Mf

Fuses but not to the extent that grain boundaries are indistinguishable

SLURRY

FBN-30 40 min 555°C-586°C
Unsoftened isotropic coal in medium moaic
Pitch coke

FBC5-30 554°C Same as FBN-30

FBC20-30 569°C
Unsoftened isotropic coal in fine (Mf) anisotropic pitch coke
additive evident - does it decrease anisotropy of pitch?

ILLN-30 50 min - 550°C
Unsoftened isotropic coal as medium (Mm) pitch coke

ILLC5-30 17 hrs @ 460°C - 1/2h @ 552°C
'A' unsoftened, isotropic coal in anisotropic (mf-Mm) pitch

1/3 hr 558°C
'B' same as 'A'

7 hrs @ 520°C - 1/5hr 650°C
'C' same as 'A' and 'B' - coal may have softened but not fused.

Additive barely noticeable

ILLC20-30
.38h @ 562°C
Unsoftened isotropic coal in anisotropic (Mf-Mm) pitch coke

PRN-30 1/4 hr. @ 408°C 'A'
Very few pores - no visible anisotropy
Low reflecance unsoftened coal and pyrite

1/2 hr. @ 506°C 'B'
Isotropic coal particles cemented in anisotropic (Mm) pitch
Large pores surrounded by anisotropic pitch coke
Coal not fused, maybe softened; pyrite framboids

PRC5-30 40 min @ 559°C
Pitch is anisotropic (Mm) but slightly larger than PRN-30B
Coal unsoftened and isotropic; no evidence of additive

PRC20-30 1/4 hr. @ 588°C

Partly softened and fused (?) isotropic coal particles

Cemented in anisotropic (Mm) coke

Coal more porous than PRN-30 and PRC5-30.

LNN-30 17 hrs. @ 350°C - 1/2 hr @ 460°C
'A' - poor polish (soft); coal is isotropic
Pitch is Mf - coal rounded (?), not really fused

3/4 hr. @ 440°C - 1/4 hr. @ 554°C

'B' - predominantly isotropic (I) at bottom - some anisotropic areas around large pores

Film layer on top of sample with spheres

Coal not as anisotropic as LNN- softened and flowed and fused but not anisotropic; at bottom, high concentration of softened partly fused isotropic coal; higher up coal is partly anisotropic (Mf); pitch is less anisotropic (Mm) than LNN- 30'C' (Mc), but more porous than LNN-30'A'

LNN-30 10 hrs. @ 480°C - 3 hrs. @ 651°C 'C'

Coal is anisotropic (Mf) as well as isotropic

Difficult to distinguish Mf coal from mosaic pitch

Bottom of sample predominantly isotropic fused, coal mass

LNC5-30 200x areas of thick solid present with large (~ 100) pores

Near top, areas of porous material (from coal?) next to non-porous material (from CLVB); separate areas of unsoftened coal particles

500x large areas of Mc along with Mm and Mf and I

Distinguishable anisotropic (Mf) coal particles surrounded by mosaics and large areas of softened but not completely fused isotropic particles

Section of anisotropic material with a solid continuous border of isotropic

Large isotropic areas predominate at bottom with few large pores but many cracks and fissures.

LNC20-30 1/2 hr. @ 552°C

Mainly isotropic at bottom and possibly the additive is in higher concentration there (Is coal settled out?) There are large areas of Mc anisotropy and Mf and I coal grains throughout the sample.

Pores usually surrounded by Mc

Can't see additive

Spheres along entire top surface of sample

LHOP 1 hr. @ 554°C

Very few pores in lower section of sample, but just above this boundary coke is much more porous

Entire sample is anisotropic (CF, FD, Mc, Mm)

LHOP'B 10 min @ 468°C

Large anisotropy (Mc) - flow mosaic and flow domain not as distinct as LHOP'A'

APPENDIX C

SIGNIFICANCE OF OPTICAL TEXTURE OF CARBONIZED COAL

Developments in the last 20 years have made use of the concept of liquid crystals to explain the development of anisotropic coke from isotropic coal melt (vitroplast). Brooks and Taylor[5] reviewed the literature and attributed this development of anisotropic coke to a distinct and recognizable phase of liquid crystals.

Marshal[6] defined three important stages: (1) the unaltered coal (principally vitrinite), showing normal anisotropy; (2) the isotropic coal (principally altered vitrinite), showing signs of having been plastic; and (3) the semicoke, characterized by minutely granular yellow/brown material. He noted that the change from (2) to (3) was abrupt and he did not observe transitional stages.

The general pattern of behaviour is described as follows. The substance melts on heating and becomes an isotropic pitch-like material of plastic or liquid consistency. An apparent exception is with the vitrinite of some low-volatile bituminous coals, where the coal does not become completely isotropic but appears to retain a preferred orientation throughout its plastic period and becomes only slightly or moderately plastic in the course of carbonization. (The anisotropic or isotropic character of a sample is the same whether it is examined in the course of heating or whether it has been subsequently cooled to room temperature before examination). No marked change occurs until the resolidification

temperature is approached, although there appears to be a progressive increase in reflectivity with temperature during the plastic period.

With rising temperatures spheres appear in the pitch-like mass. With increase of temperature, and to a certain extent with time at a fixed temperature, the spheres grow larger. When they are of micron size they can be detected, with strong illumination and crossed nicols (crossed polarizing filter), with the optical microscope. In thermally metamorphosed coals such spheres may occur within the vitrinite but never within the inertinite macerals.

At an advanced stage during the resolidification from the liquid state the spheres will have replaced a large part of the plastic sample and begun to interfere with one another's further enlargement with progressive increase in temperature. At this point a mosaic begins to form, as the remaining isotropic material is converted to the substance of which the spheres were comprised. When no more isotropic material of pitch-like consistency remains and only anisotropic material, or mesophase, is present, the mosaic structure is complete and then, or shortly afterward, the mesophase solidifies into semi-coke.

The formation of the mosaic begins with the coalescence of the spheres. However, if the coalescence of spheres becomes general, many new connections are formed and the merging to form larger spheres cannot occur. This process goes to completion with the conversion of interstitial pitch to mesophase. Since coalescence is occurring in three dimensions, a structure is formed in which mesophase, as seen in any plane, comprises

areas in which the orientation is fairly uniform, connected to one another by zones of mesophase in which the lamellae curve around sharply to conform to the orientation of the next fairly uniform area. Thus, although the term "mosaic" has been employed the structure is not composed of sharply bound units. At a slightly higher temperature the mesophase changes to solid semicoke without obvious change of structure. With vitrinite these events occur within a temperature range of a few degrees.

The size of the anisotropic regions (mosaic units) developed by vitrinite in coking coals was observed to be related to rank. Thus bituminous coal of the highest volatile-matter yield gives rise to coke which appears isotropic in the optical microscope but which contains submicroscopic ordered domains analagous to the microscopically observed mosaic units in coke from other coals. As the rank of the coal increased, the size of the mosaic units in the coke increased to the stage of medium-volatile bituminous coals. At this point it reached a maximum; the vitrinite in low-volatile coal yielded a coke having fine domains but exhibiting in addition larger-scale aggregate polarization properties which were absent with cokes from high-volatile coals. The larger-scale effect was seen to be related to the preferred orientation originally present in the coal.

The more plastic the coal became during carbonization between 350 and 500°C, the larger the anisotropic regions - mosaic units - were observed to be. The microscopic texture of coke does not alter significantly after solidification on heating up to 1500-1700°C and the form and size of

domains persist, although the degree of anisotropy increases progressively with increasing temperature.

Therefore, to determine the resolidification point of a coking coal during plastofrost carbonization several observations must be made.

Firstly, the coal must be observed to have been plastic. For most coals, the vitrinite, which in its unaltered state was anisotropic, becomes isotropic. For some low-volatile coals vitrinite anisotropy is not completely lost, however, evidence of the plastic phase should be observed.

Secondly, spheres should appear with increasing temperature. Since these spheres coalesce up to the resolidification temperature they should grow and possibly form large (up to 1mm) regions of uniform orientation.

Finally, since the shape and size of the anisotropic domains does not change appreciably above the resolidification point, the point at which these domains cease to grow and change should be the resolidification temperature. At this point the conversion of pitch to mesophase has been completed and the mesophase has solidified to form semicoke.

SUBCONTRACTOR'S STATEMENT

TECHNICAL PROPOSAL

A. Scope and Objectives

1. Plastofrost Measurements

Measurements are to be undertaken on 12 coal samples and 12 coal-oil-slurry samples. These measurements and others specified in the Bidder's Invitation are intended to assess coking propensity, that is, the suitability of the source material for the production of metallurgical coke.

The plastofrost procedure is not a widely used rheological method (such as dilatometry and plastometry). It is an experimental technique. Thus, it seems worthwhile to describe the technique and justify its application to coal-oil-slurries.

The procedure consists of arranging thermocouples axially in a column of compacted coal. The column is then heated at a uniform rate from the bottom for an appropiate time to fully coke the bottom of the column but leave the top uncoked. Temperatures are recorded with time. After cooling, the column is cut apart and the central core is polished for microscopic examination. It is possible to associate then the appearance of pores or bubbles in the coal grains, grain smoothing, fusion, bubble expansion, completion of fusion and the onset of coke anisotrophy with temperature since the temperature time history of the column was measured. Not only can the coal-to-coke transformation be seen for the coal sample, rheological properties such as the plastic range, onset of softening (contraction), onset of bubble or pore growth (dilatation) are also measured. The plastofrost gives, therefore, much more information than other rheological techniques such as dilatometry or plastometry.

In addition to the wealth of information the procedure gives on the coal-to-coke transformation, there is a further, compelling reason to use the method. CANMET coprocessing studies have used both Appalachian and Western Canadian coals. It is to be expected, therefore, that coals from both regions will be among the samples received for measurement. Unfortunately, the standard rheological techniques of dilatometry and plastometry do not adequately assess the coking propensity of Western Canadian bituminous coals (Grandson, Pierce and Price, Energy Research Laboratory Report, ERP/EPL 78-104 (TR), CANMET (1978)). Many Western Canadian coking coals show total dilatation which are so low that the coal would be classed as non coking if it were of Appalachian origin. Studies done in the Coal Research Laboratory at the University of Waterloo show that this does not arise with the plastofrost. The plastic range determined by plastofrost procedure, for example, are comparable for Appalachian and Western Canadian coals (Duever, Silveston and Readyhough, "Application of a modified plastofrost apparatus to coal rheology", MS submitted to the C.I.M. Bulletin, 1986). Indeed, the Coal Research Laboratory first became interested in the use of the plastofrost through DSS contract work undertaken by the Laboratory for CANMET. This work investigated the reasons for the failure of dilatometry and plastometry with the Western Canadian

coals.

Justifications for using the plastofrost procedure, then, are: 1) it permits data for all coals used by CANMET to be correlated together (otherwise, with dilatometry, Appalachian and Western Canadian coals would have to be analyzed separately), 2) it provides much more rheological information than other rheological techniques. Possibly, the visualization the procedure provides on the coal-to-coke transformation maybe useful in understanding mechanisms for formation of residual solids in the CANMET process.

2. Surface Area and Pore Size Distribution

Measurements are to be made on the 24 samples mentioned in 1) above. These measurements are of questionable value. Although, surface area and pore size distribution are useful in coke characterization where they are important parameters for coke reactivity, these measurements are very difficult to interpet for coal. Surface area is measured by nitrogen adsorption at liquid nitrogen temperature, but at this temperature, nitrogen diffusion is activated and equilibrium is reached only after many days. It is possible to arbitrarily measure adsorption after one or two hours, but such data is useful only to evaluate the effect of coal treatment and even then interpetation is not reliable. The presence of an oil (for the coal-oil-slurries) will interfere further with nitrogen diffusion, not to mention the havoc it would play with the high vacuum systems needed for the measurement technique. Surface area, but not pore size distribution, can be determined by swelling with solvents such as benzene. However, the same difficulties mentioned above for nitrogen adsorption occur.

Pore size distribution is measured by nitrogen adsorption for micropores and mercury penetration for the macropores. The later procedure gives unreliable data for coal because of deformation of the coal solid. Coal is wetted by oil, so the macropore volume would be negligible for coal-oil-slurries.

In view of the above problems, we do not recommend a full set of measurements. Instead, we propose a set of six exploratory ones to see if useful information can be obtained. These would be restricted to coal samples. If useful information indeed appears, further measurements could be added.

B. Methodology Proposed

1. Plastofrost Measurements

We have assumed that CANMET will undertake sample preparation. Coals are heterogeneous minerals so that devising representative samples is a major concern. Adequate replication of measurements is essential. Furthermore, oxidation is a problem with all rheological procedures including the plastofrost one. Thus, we assume that the 24 samples requested provide for replication. As the plastofrost procedure is still experimental, it will be necessary to test its reproducibility and we address this below. All samples to be sent to Waterloo must be placed in nitrogen filled vacuum jars immediately after preparation. They should be

stored in a cold chamber prior to shipment. Shipment should be by overnight courier.

About 75 g of sample are needed for each plastofrost measurement. We will need at least 250 gm of each sample allowing for wastage and accident. For the coal-oil-slurries, 500 gm samples will be needed so as to allow for reproducibility studies.

Reproducibility of plastofrost measurements should be settled by a DSS Contract to the Waterloo Coal Research Laboratory which has been approved, but not yet funded. Nevertheless, the use of coal-oil-slurries will represent a new situation so we propose to add a further 4 replicate measurements to the 24 indicated in the Bid Invitation.

Procedures to be used are storage of all samples received in a cold chamber. Samples will be opened just prior to use. For the plastofrost, the first task is to mount thermocouples in their ceramic holders which are then placed in the coking cylinder. The coal sample is then packed into the cylinder. This is a painstaking task requiring several hours. Coking requires about 2 1/2 hours. Cooling is done overnight. After the sample is removed, it is impregnated with resin. Once the resin has hardened (overnight), the column is sawed in half, perpendicular to the thermocouples, and each half is ground and polished using special wheels and abrasives. This operation requires most of a day. The final task is microscopic examination. This takes several hours. The correlation of observations and temperature is not a trivial job.

We assume that the coal-oil-slurries will be solids at ambient temperatures so we do not anticipate handling problems. The high asphaltene content may change the appearence of the coke in the various stages of the coal-to-coke transformation. Thus, some observations, such a grain rounding, may not be possible.

The bottom layer in the column will be coked and would be suitable for surface area and pore size distribution measurements. Samples will be retained for this purpose, but measurements will not be made.

2. Surface Area and Pore Size Distribution

Only several grams of sample are needed for measurements and aging has a negligible effect. Samples for study will be taken from the plastofrost samples.

Nitrogen adsorption (B.E.T.) and mercury penetration are standard techniques with ASTM procedures. The only problems are that devolatilization occurs to a minor extent on degassing. Thus, equipment cleaning is needed. As discussed in the previous section, coal-oil-slurries cannot be used.

CYCLONE ENGINEERING
SALES LTD.

CONTRACT SERIAL NUMBER 69139-01-SQ

DSS REQUISITION NUMBER 23440-6-9139

CONTRACTOR Cyclone Engineering Sales Ltd.

FINAL REPORT
Prepared for
DSS 23440-6-9139101ISQ
by
David E. Pearson & Associates Ltd.
(Petrographic Characterization)

MICROMEDIA

CYCLONE ENGINEERING SALES LTD.
9719 - 60 Avenue
Edmonton, Alberta
T6E 0C4

Telephone: (403)436-1385
Telex: 037-3793
Fax: (403)436-4103

Petrographic Characterization

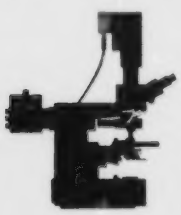
of

Treated and Untreated Coals.

May, 1987.

Prepared for

CANMET.



David E. Pearson & Associates Ltd.

Consulting Coal Geologists & Petrographers,

804 Leota Place,

Victoria,

British Columbia, V8Y 1H2, Canada.

David E. Pearson & Associates Ltd.
Consulting Coal Geologists & Petrographers

804 Leota Place, Victoria, B.C. V8Y 1H2 (604) 658-5963
Facsimile (Canon 520): (604) 658-1029

May 8, 1987.

Mr. Vipul Patel,
Cyclone Engineering Sales Ltd.,
9719 - 60th Avenue,
Edmonton, Alberta.
T6E 0C4

Dear Mr. Patel:

Re: Petrographic Characterization of CANMET's Coal Samples.

We are pleased to provide you with our report on the characterization of the suite of treated and untreated coals from CANMET. We trust your client will find the information to be most useful.

We thank you for the opportunity to have been of assistance to you.

Yours very truly,
David E. Pearson & Associates Ltd.,

PROFESSIONAL ENGINEER
PROVINCE OF BRITISH COLUMBIA
DAVID E. PEARSON

David E. Pearson, Ph.D., P.Eng.

SUMMARY

Twelve coal samples have been microscopically characterized in white polarized light and their fluorescent properties examined.

The samples represent a suite of four high volatile coals in a natural condition, and after treatment with various amounts of ferrous sulphate.

Although the samples were prepared under isopropanol, careful examination of high-resolution photomicrographs reveals no traces of the iron compound.

The absence of, or inability to recognize the ferrous sulphate deposits, using the optical equipment used, is discussed.

INTRODUCTION

Twelve samples of crushed coal were received at the Coal Laboratory on March 2, 1987. The samples were identified as follows:-

Cyclone Engineering Sales Ltd., Samples DSS#1 to DSS #12.

In a subsequent phone conversation with Dr. Forda, it was revealed that there were three sets of four samples, and that two of the sets had been treated with ferrous sulphate. Consequently, the specific request was made to prepare the samples under isopropyl alchohol, and to carefully look for traces of the iron compound.

SAMPLE PREPARATION

Each coal sample was coned and quartered and reduced to provide sufficient material for two pellets. One portion of the coal was then mixed with a lucite thermoplastic and pelletized under slight pressure at a temperature of 100°C. The other portion of coal was mixed with a resin and allowed to cure under ambient conditions. Both sets of pellets were subsequently ground and polished under isopropanol using Buehler equipment.

PETROGRAPHIC EXAMINATION

The polished samples were examined under an oil-immersion lens using a Leitz Orthoplan MPV Compact microscope-photometer. The control panel of the microscope and a Swift Automatic Point Counter are interfaced to a Hewlett-Packard 85 microcomputer, which captures the data. These data are then passed to a Hewlett-Packard 9816 microcomputer, an Apple LaserWriter printer and a Hewlett-Packard 7475A plotter for processing, printing and draughting of results. Photomicrographs were taken under oil-immersion in plane-polarized white light.

The reflectance of one hundred individual vitrinite 'A' grains was measured in the rank analyses. Standardization of photometer-readout was performed before and after the analysis, if necessary. Maximum reflectance values were retained by the computer.

One thousand grains were counted in the maceral analyses, at a traverse interval of 0.5mm. During the maceral analyses, the random reflectance of each maceral was also determined, in polarized light, and these 1000 values were used to construct the reflectogram.

For characterization of the fluorescence properties, a Zeiss Universal microscope-photometer equipped with a scanning monochrometer was used. This equipment is controlled by an Intercolour Zeiss-Zonax microcomputer. Photomicrographs were taken using blue light excitation at 450-490nm, and a barrier filter at 515nm, under oil immersion.

RESULTS

The results of all analyses are contained as Appendices, and are shown in the **Summary of Petrographic Analysis.**

For these samples the reported results are:-

* Reflectance values
* Reflectance statistics
* Vitrinite-type histogram
* 1000 Maceral counts
* Maceral statistics
* Maceral-distribution diagram
* 1000 - point reflectogram
* Predicted Strength & Balance Index
* Predicted D.I. 30/15
* Predicted coke strength
* Predicted free swelling index

DISCUSSION

The petrographic analyses of the untreated coals show them to be normal high volatile coals, all of which have high concentrations of the reactive macerals vitrinite, and, to a lesser extent, exinite. The fluorescence characteristics are consistent with the rank of the coals and the chemical nature of the constituents. Photomicrographs confirm these conclusions.

The absence of any evidence for the presence of ferrous sulphate is also notable, and begs the question, where is it? Study of the photomicrographs of DSS Samples #9 - #12 (those treated with the largest quantity of salts), show

no peripheral salt deposits.

If the salt deposits are less than 0.5µm, then they are beneath the resolution of the optical microscope used. Alternatively, sulphates generally possess a hardness of <4, and often <2, and some salts rarely form crystals. There is a possibility therefore, that the ferrous sulphate may coat individual grains but because of its Mohs' hardness it may not have survived polishing despite the use of isopropanol.

Finally, there is the likelihood that the refractive index of ferrous sulphate may be so similar to that of the immersion oil (Ne@25°C=1.5178), that its outline would disappear (Becke line effect), and its reflectance would be the same as the mounting medium.

Despite our best efforts, the recognition of ferrous sulphate has eluded us, we think for one, or all, of the reasons described above.

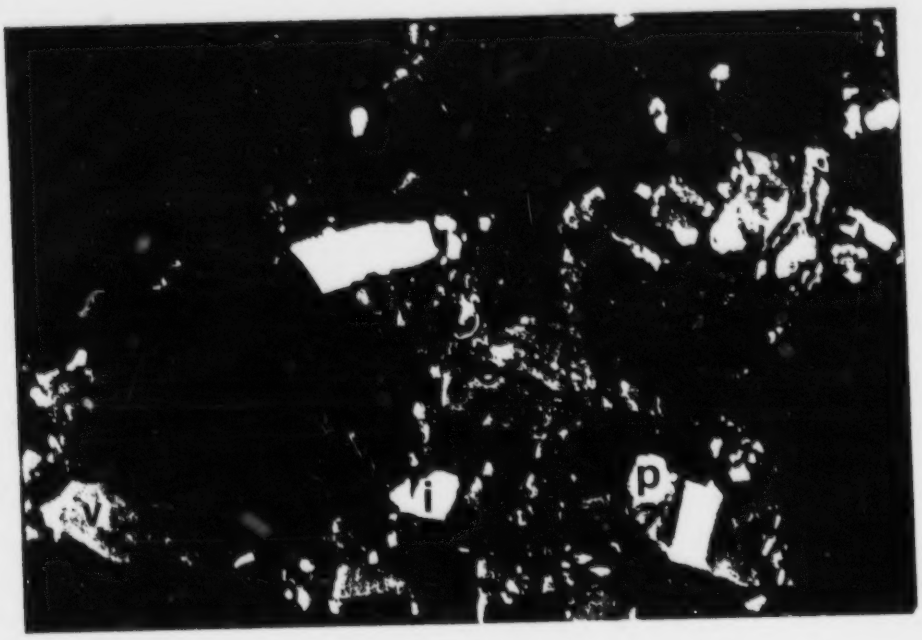


Plate 1. Sample DSS #12. Grains of Vitrinite (v), Inertinite (i), black Exinite in Vitrinite at (e), and bright Pyrite (p). Diameter of outer circle at the centre of the photomicrograph is 10µm.

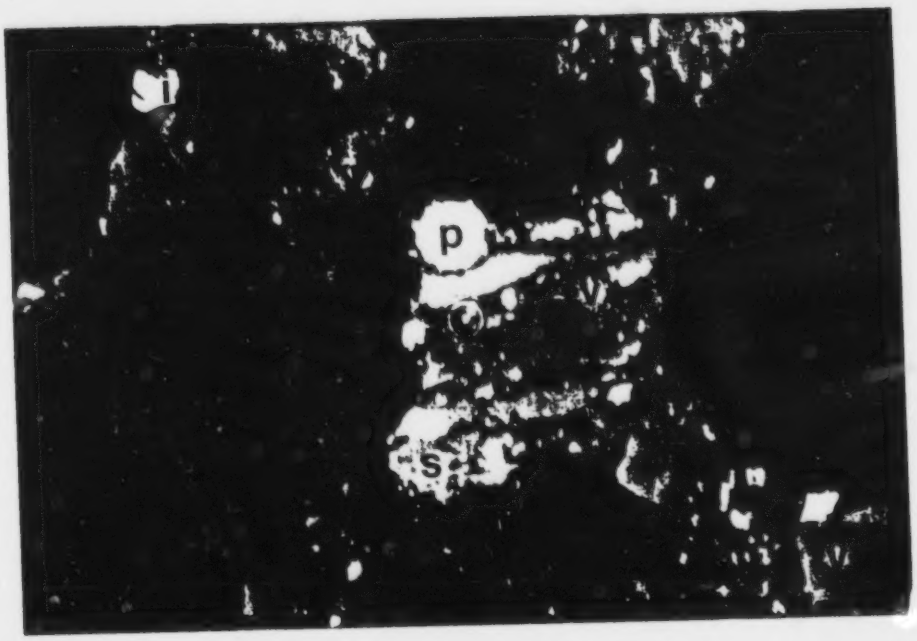


Plate 2. Sample DSS #12. Grain of Vitrinite (v),with semifusinite(s), and bright Pyrites (p). Diameter of outer circle at the centre of the photomicrograph is 10µm.

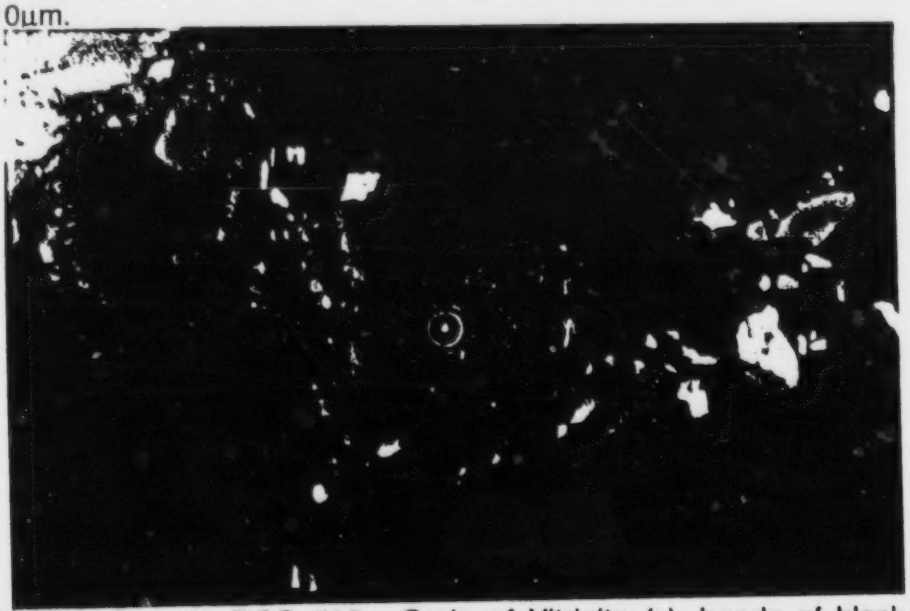


Plate 3. Sample DSS #12. Grain of Vitrinite (v), bands of black Exinite in Vitrinite at (e). Diameter of outer circle at the centre of the photomicrograph is 10µm.

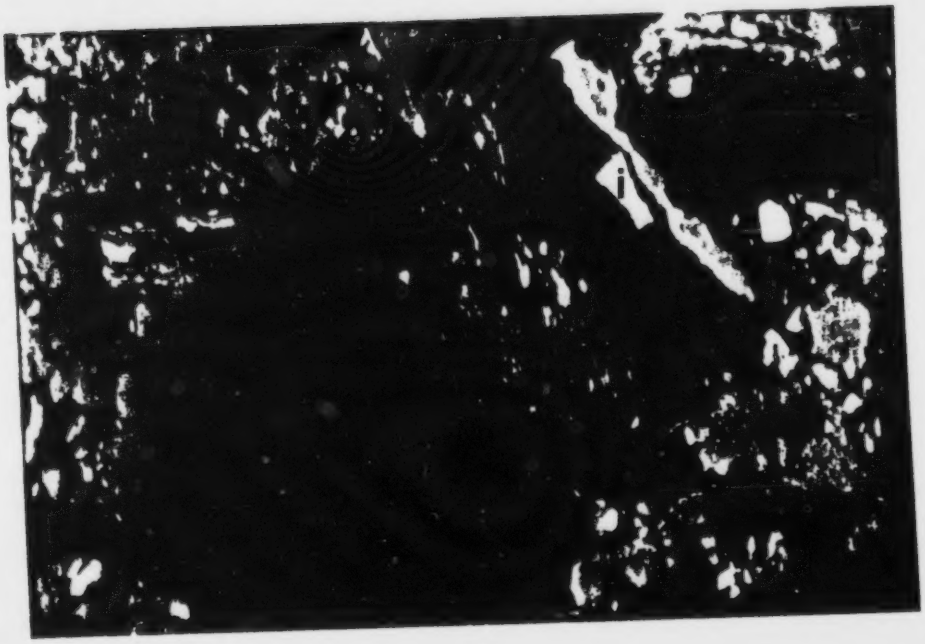


Plate 4. Sample DSS #12. Large banded composite grain of Vitrinite (v), with Inertinite (i), black Exinite in Vitrinite at (e). Diameter of outer circle at the centre of the photomicrograph is 10µm.

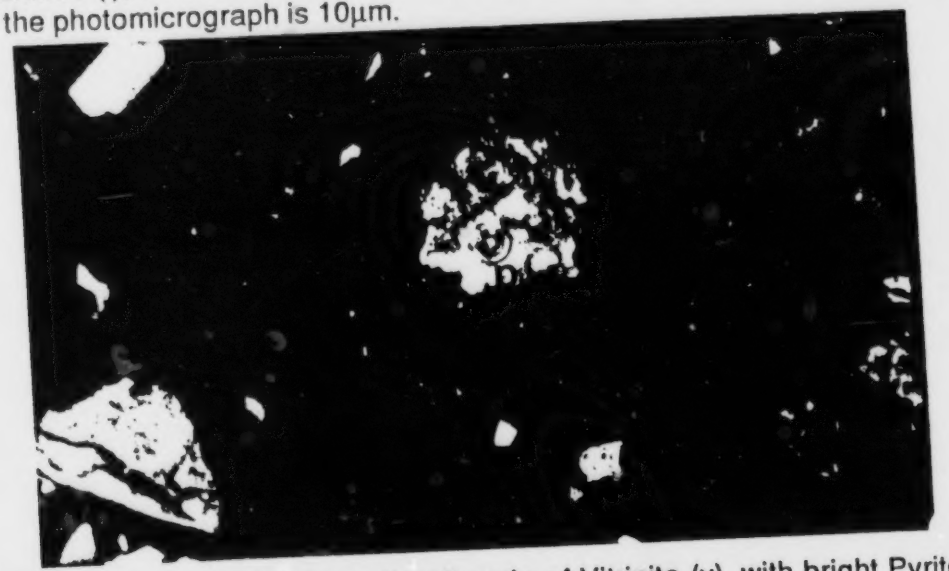


Plate 5. Sample DSS #12. Small grain of Vitrinite (v), with bright Pyrite (p) at the margin and inside the grain. Diameter of outer circle at the centre of the photomicrograph is 10µm.

Plate 6. Sample DSS #12. Grain of Vitrinite (v), with thin pyrite vein(p), and Inertinite (i). Diameter of outer circle at the centre of the photomicrograph is 10μm.

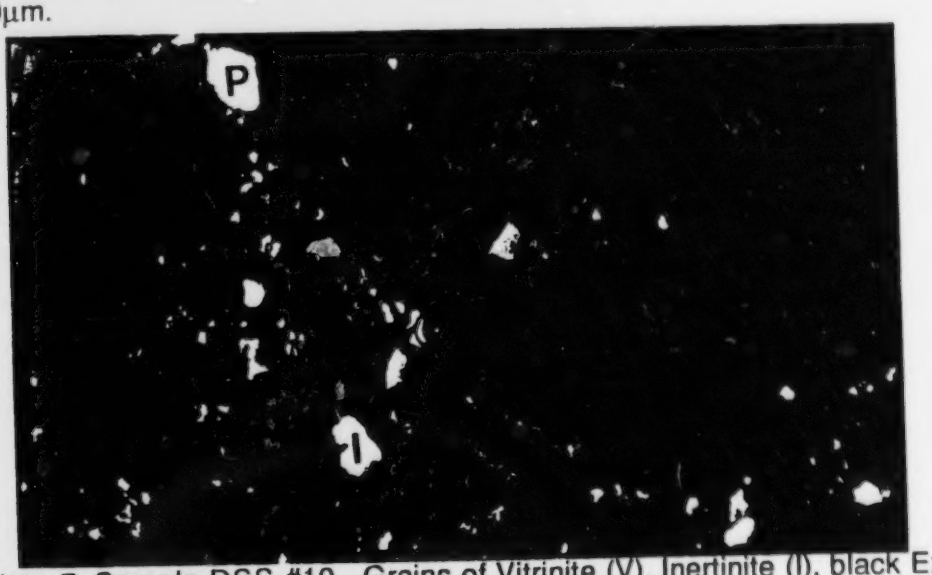


Plate 7. Sample DSS #10. Grains of Vitrinite (V), Inertinite (I), black Exinite in Vitrinite at (E), and bright Pyrite (P). Diameter of outer circle at the centre of the photomicrograph is 10μm.

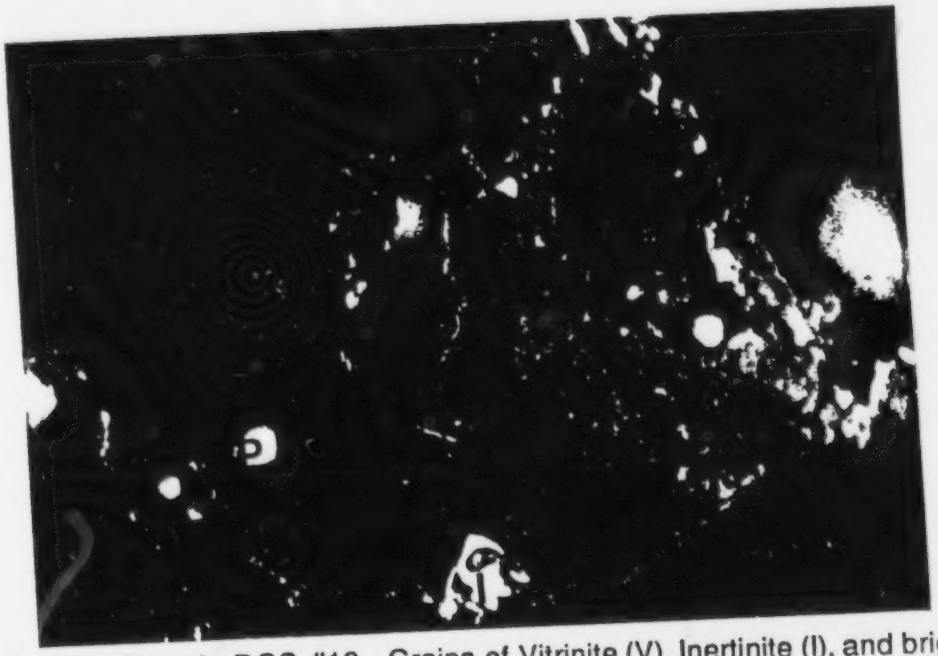


Plate 8. Sample DSS #10. Grains of Vitrinite (V), Inertinite (I), and bright Pyrite (P). Diameter of outer circle at the centre of the photomicrograph is 10µm.

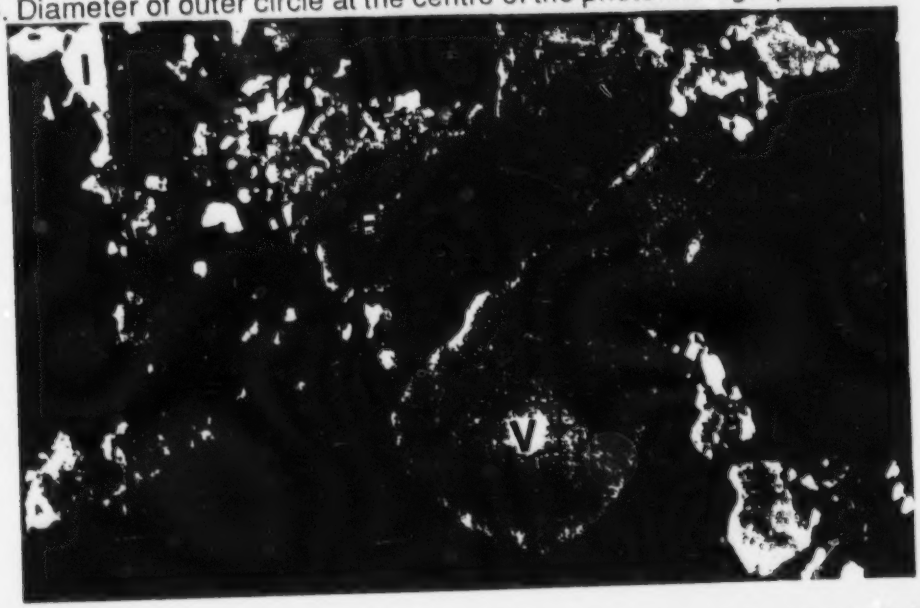


Plate 9. Sample DSS #11. Grains of Vitrinite (V), Inertinite (I), black Exinite in Vitrinite at (E). Diameter of outer circle at the centre of the photomicrograph is 10µm.

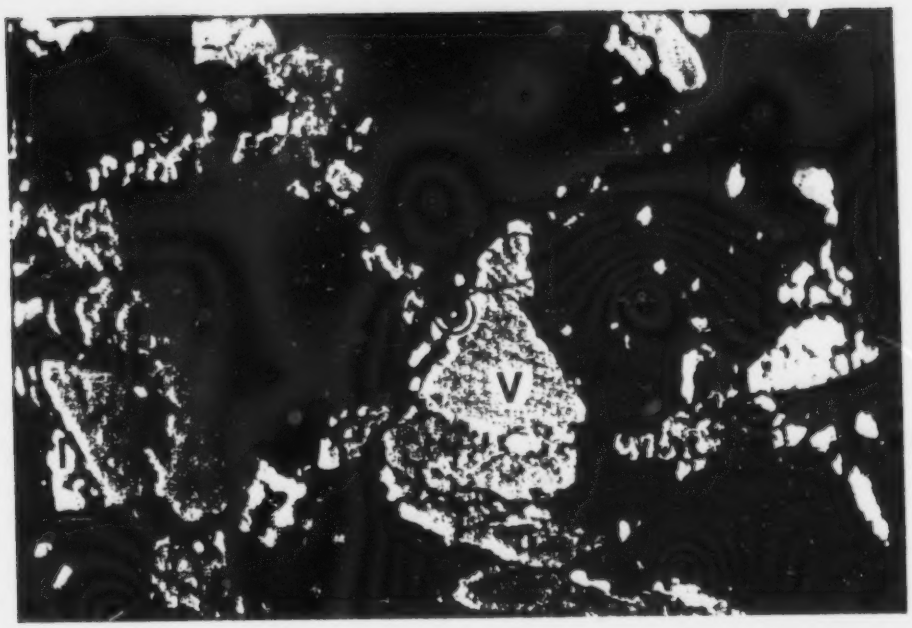


Plate 10. Sample DSS #9. Grains of Vitrinite (V), Inertinite (I). Diameter of outer circle at the centre of the photomicrograph is 10µm.

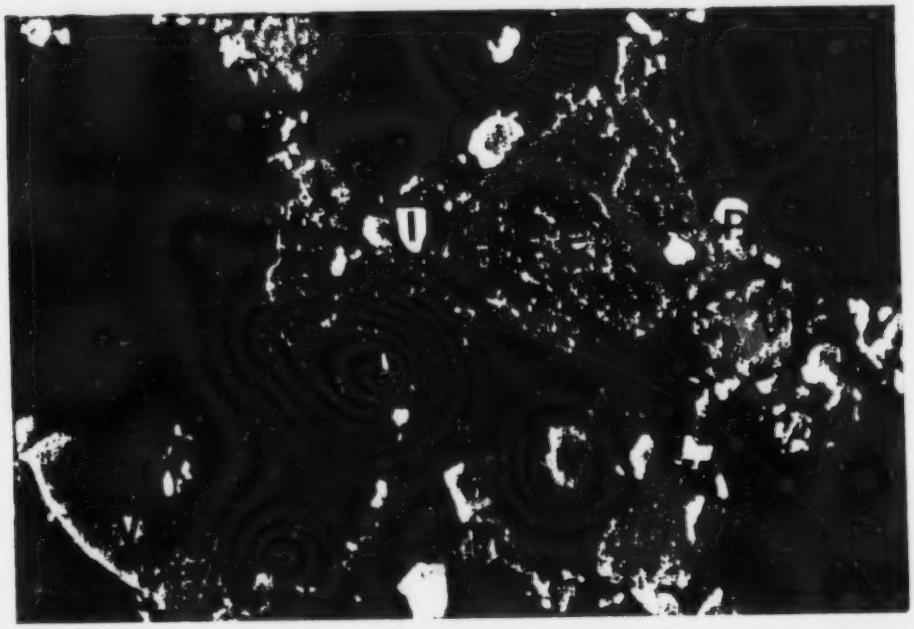


Plate 11. Sample DSS #9. Grains of Vitrinite (V), Inertinite (I), black Exinite in Vitrinite at (E), and bright Pyrite (P). Diameter of outer circle at the centre of the photomicrograph is 10µm.

Plate 12. Lingan Sample. Grains of Vitrinite (V), Inertinite (I, black Exinite in Vitrinite at (E). Diameter of outer circle at the centre of the photomicrograph is 10μm.

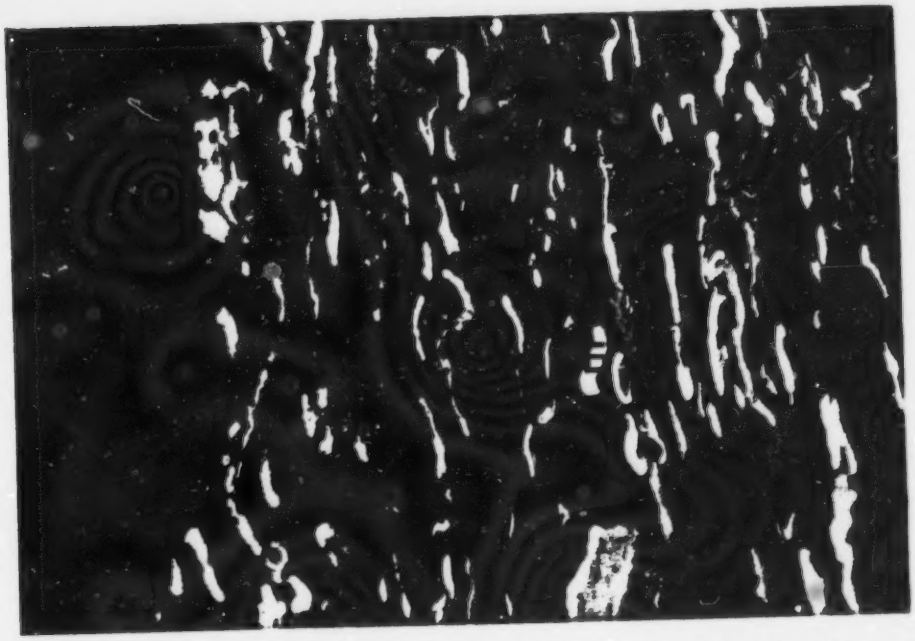


Plate 13. Lingan Sample. Same view as above, but showing microspore (exinite) fluorescence. Diameter of outer circle at the centre of the photomicrograph is 10μm.

Plate 14. Prince Sample. Grains of Vitrinite (V), Inertinite (I), black Exinite in Vitrinite at (E). Diameter of outer circle at the centre of the photomicrograph is 10μm.

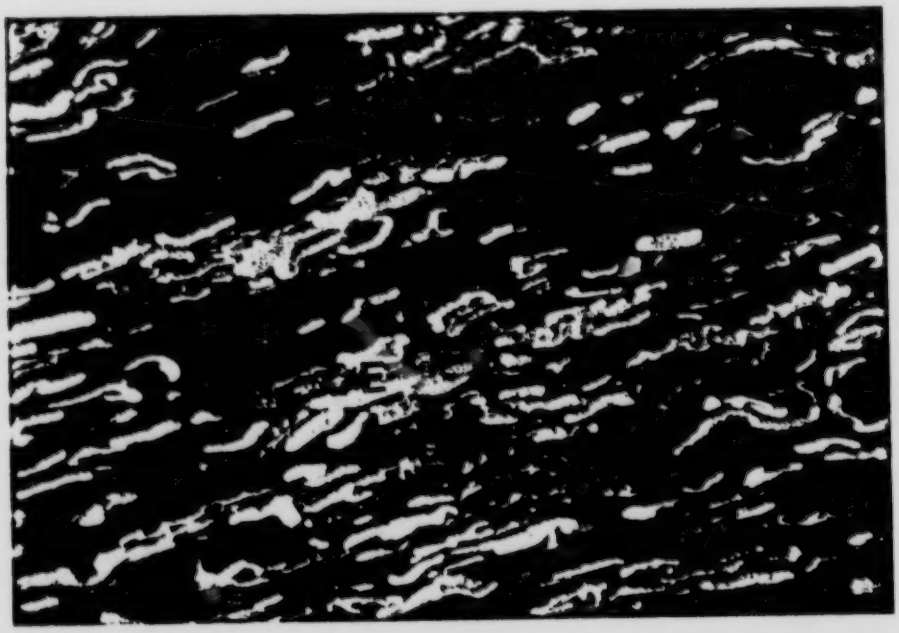


Plate 15. Prince Sample. Same view as above, but showing microspore (exinite) fluorescence. Diameter of outer circle at the centre of the photomicrograph is 10μm.

SAMPLE IDENTIFICATION

Laboratory number M4881
Untreated Prince Mine
Ash 7.00% Sulphur 0.80%

PETROGRAPHIC INDICES

Mean Maximum Reflectance	%	0.76
Composition Balance Index		0.74
Calculated Strength Index		2.83
Calculated Stability Index		29
Estimated Coke Strength DI 30/15		85.0
Predicted Free Swelling Index		5

DISTRIBUTION OF VITRINITE TYPES

V - 6	%	7
V - 7	%	78
V - 8	%	14
V - 9	%	1

REACTIVE COMPONENTS

Vitrinite	%	67.4
Exinite	%	6.9
Reactive Semifusinite	%	6.2
Total Reactives	%	80.5

INERT COMPONENTS

Macrinite	%	5.9
Inert Semifusinite	%	7.2
Fusinite	%	1.2
Inertodetrinite	%	1.2
Mineral Matter	%	4.0
Total Inerts	%	19.5

SAMPLE IDENTIFICATION

Laboratory number M4882
Untreated Lingan Mine
Ash 7.00% Sulphur 0.80%

PETROGRAPHIC INDICES

Mean Maximum Reflectance..... %	0.98
Composition Balance Index.....	0.52
Calculated Strength Index.....	3.55
Calculated Stability Index.....	42
Estimated Coke Strength DI 30/15.....	91.0
Predicted Free Swelling Index.....	8

DISTRIBUTION OF VITRINITE TYPES

V - 8.....%	5
V - 9.....%	51
V - 10.....%	43
V - 11.....%	1

REACTIVE COMPONENTS

Vitrinite.....%	70.1
Exinite.....%	6.0
Reactive Semifusinite.....%	6.7
Total Reactives.....%	82.8

INERT COMPONENTS

Macrinite.....%	3.8
Inert Semifusinite.....%	8.1
Fusinite.....%	0.9
Inertodetrinite.....%	0.4
Mineral Matter.....%	4.0
Total Inerts.....%	17.2

SAMPLE IDENTIFICATION

Laboratory number M4780
DSS #1 - Forestburg
Ash 6.00% Sulphur 0.50%

PETROGRAPHIC INDICES

Mean Maximum Reflectance	%	0.53
Composition Balance Index		0.49
Calculated Strength Index		2.45
Calculated Stability Index		0
Estimated Coke Strength DI 30/15		0
Predicted Free Swelling Index		0

DISTRIBUTION OF VITRINITE TYPES

V - 4	%	29
V - 5	%	62
V - 6	%	9

REACTIVE COMPONENTS

Vitrinite	%	82.7
Exinite	%	2.2
Reactive Semifusinite	%	3.9
Total Reactives	%	88.8

INERT COMPONENTS

Macrinite	%	2.6
Inert Semifusinite	%	1.3
Fusinite	%	1.3
Inertodetrinite	%	2.6
Mineral Matter	%	3.4
Total Inerts	%	11.2

SAMPLE IDENTIFICATION

Laboratory number M4780
DSS #2 - Illinois #6
Ash 7.00% Sulphur 0.90%

PETROGRAPHIC INDICES

Mean Maximum Reflectance	%	0.55
Composition Balance Index		0.61
Calculated Strength Index		2.52
Calculated Stability Index		13
Estimated Coke Strength DI 30/15		73.3
Predicted Free Swelling Index		<<4

DISTRIBUTION OF VITRINITE TYPES

V - 4	%	13
V - 5	%	73
V - 6	%	13
V - 7	%	1

REACTIVE COMPONENTS

Vitrinite	%	83.3
Exinite	%	1.0
Reactive Semifusinite	%	1.9
Total Reactives	%	86.2

INERT COMPONENTS

Macrinite	%	4.9
Inert Semifusinite	%	3.0
Fusinite	%	1.0
Inertodetrinite	%	0.9
Mineral Matter	%	4.0
Total Inerts	%	13.8

Vitrinite Reflectance

CANMET SAMPLES
Untreated Prince Mine Sample

BASIC STATISTICS

Total Number of Observations	100
Mean Maximum Reflectance (Romax)...%	.76
Standard Error of the Mean	0.00
Coefficient of Variation...%	6.02
Variance	.0021
Standard Deviation	.0456
Skewness	.4921
Kurtosis	4.4009

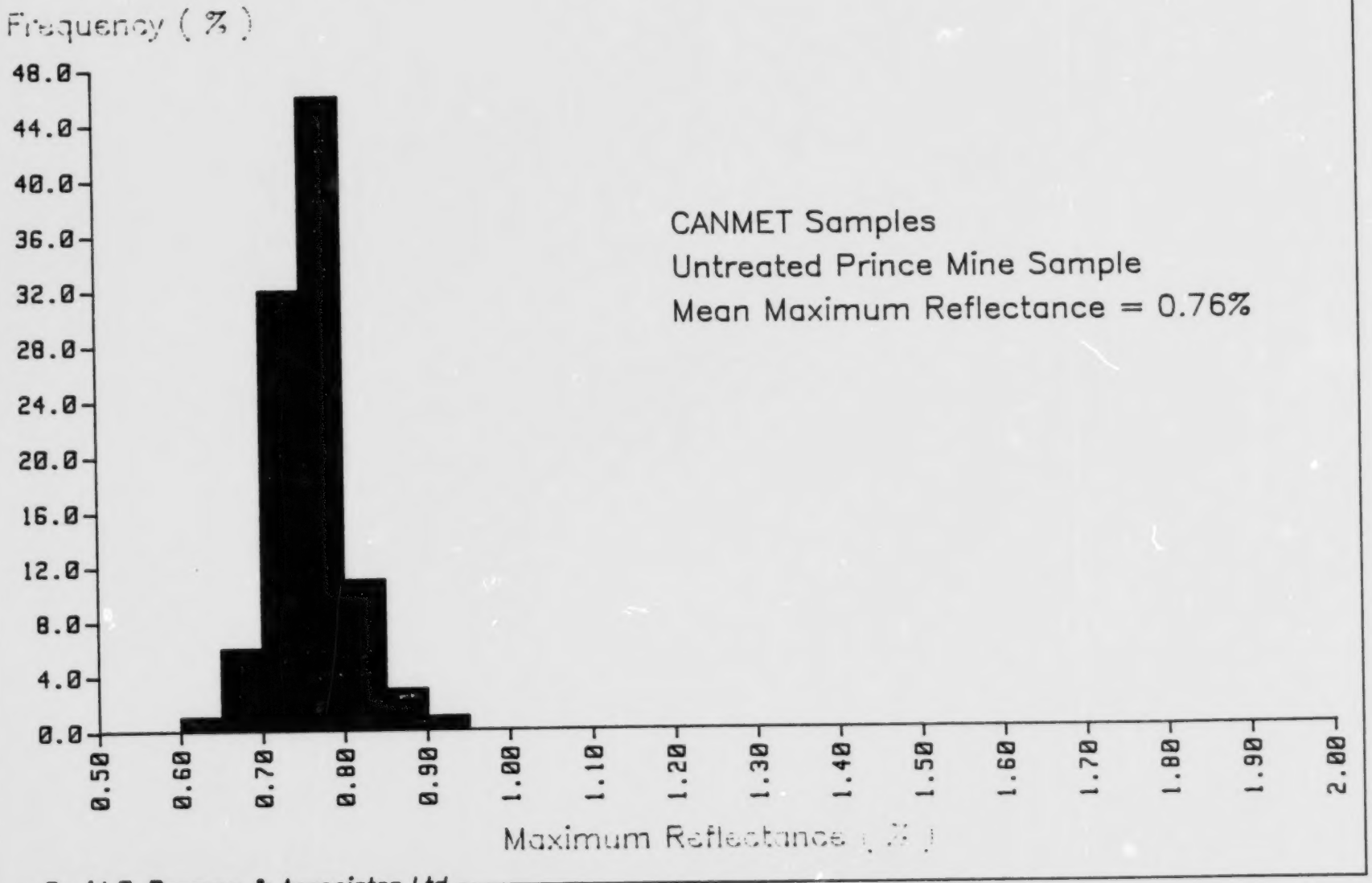
CELL STATISTICS

Cell Number	Lower Limit	Number of Observations	Frequency (%)
3	0.60	1	1.00
4	0.65	6	6.00
5	0.70	32	32.00
6	0.75	46	46.00
7	0.80	11	11.00
8	0.85	3	3.00
9	0.90	1	1.00

VITRINITE TYPE DISTRIBUTION

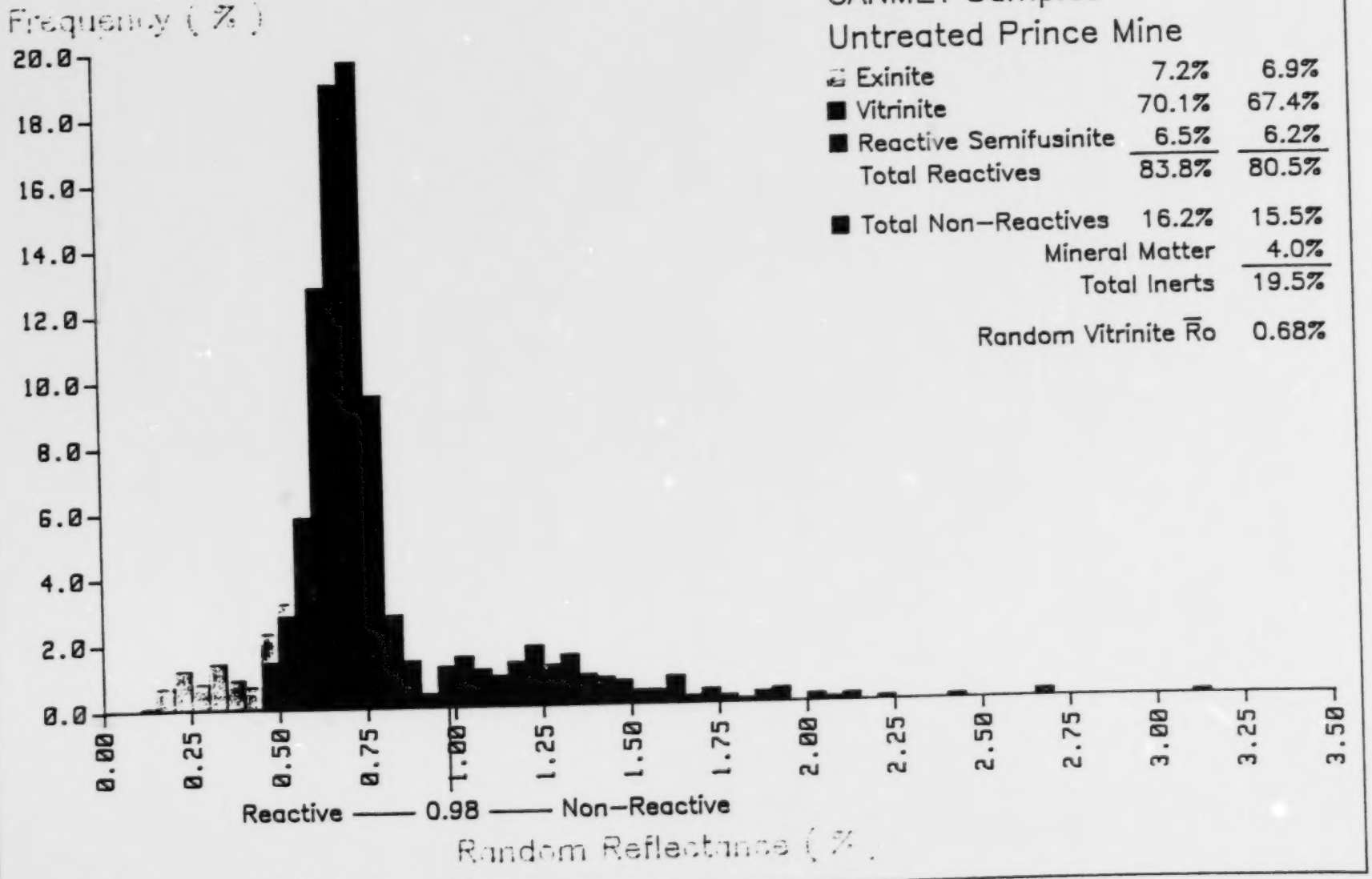
Vitrinite Type	Frequency (%)
V6	7.00
V7	78.00
V8	14.00
V9	1.00

VITRINITE HISTOGRAM



David E. Pearson & Associates Ltd.

REFLECTOGRAM



CANMET Samples

Untreated Prince Mine

Exinite	7.2%	6.9%
Vitrinite	70.1%	67.4%
Reactive Semifusinite	6.5%	6.2%
Total Reactives	83.8%	80.5%
Total Non-Reactives	16.2%	15.5%
Mineral Matter		4.0%
Total Inerts		19.5%
Random Vitrinite $\overline{R}o$		0.68%

David E. Pearson & Associates Ltd.

Maceral Analysis

CANMET SAMPLES
Untreated Prince Mine

Count #	1	2	3	4	5	6	7	8	9	10
Vitrinite	69	74	71	75	76	71	62	66	64	73
Exinite	11	7	8	4	5	5	9	9	4	10
Reactive Semifusinite	2	7	4	9	7	12	6	6	9	3
Total Reactives	82	88	83	88	88	88	77	81	77	86
Macrinite	6	3	10	7	7	5	8	3	6	6
Inert Semifusinite	6	7	4	5	2	6	11	14	16	4
Fusinite	2	1	0	0	2	0	3	2	1	2
Inertodetrinite	4	1	3	0	1	1	1	0	0	2
Total Non-Reactives	18	12	17	12	12	12	23	19	23	14

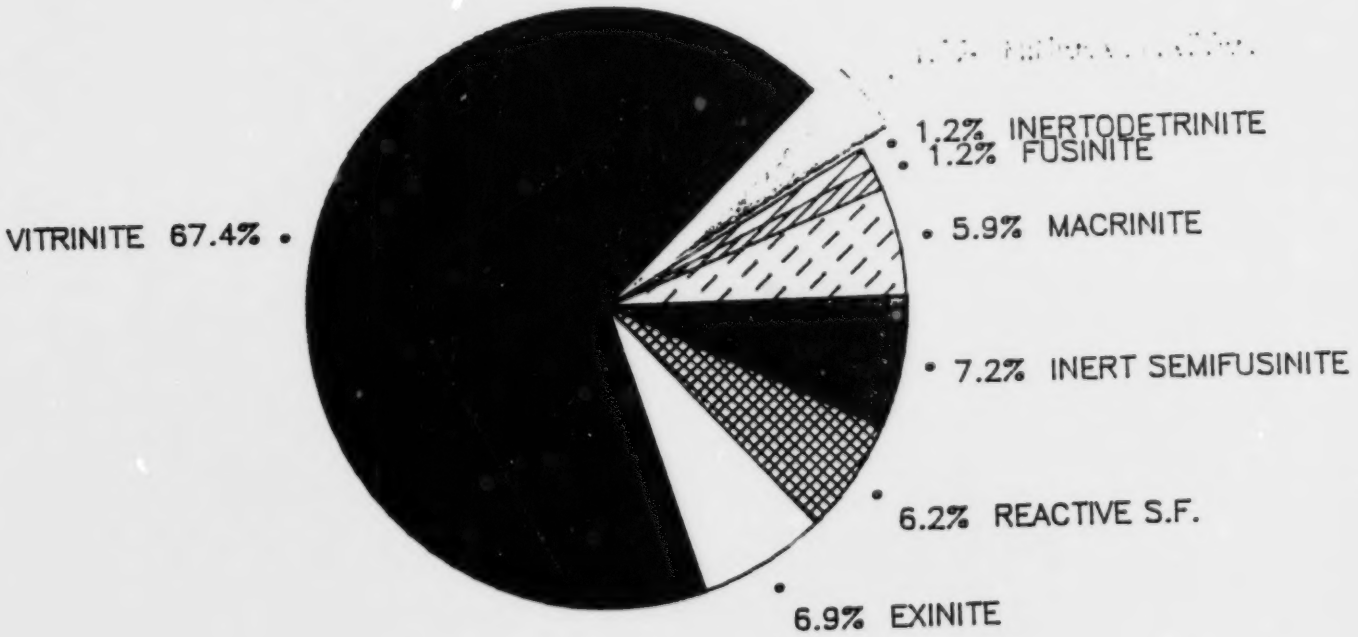
Basic Statistics	Mean	St.Deviation	Variance
Vitrinite	70.1	4.8	22.8
Exinite	7.2	2.6	6.6
Reactive Semifusinite	6.5	3.0	9.2
Total Reactives	83.8	4.5	20.0
Macrinite	6.1	2.1	4.5
Inert Semifusinite	7.5	4.6	21.4
Fusinite	1.3	1.0	1.1
Inertodetrinite	1.3	1.3	1.8
Total Non-Reactives	16.2	4.5	20.0

Maceral Data Corrected For Mineral Matter Content

Vitrinite	67.4
Exinite	6.9
Reactive Semifusinite	6.2
Total Reactives	80.5
Macrinite	5.9
Inert Semifusinite	7.2
Fusinite	1.2
Inertodetrinite	1.2
Total Non-Reactives	15.5
Mineral Matter	4.0
Total Inerts	19.5

MACERAL DISTRIBUTION

Semifusinite – Pearson Method



CANMET Samples
Untreated Prince Mine

David E. Pearson & Associates Ltd.

Coke Stability Calculation

CANMET SAMPLES
Untreated Prince Mine

Coke Stability Index Calculation

Total Inerts = 19.5 %

(100 - TI) / 100 = 0.81

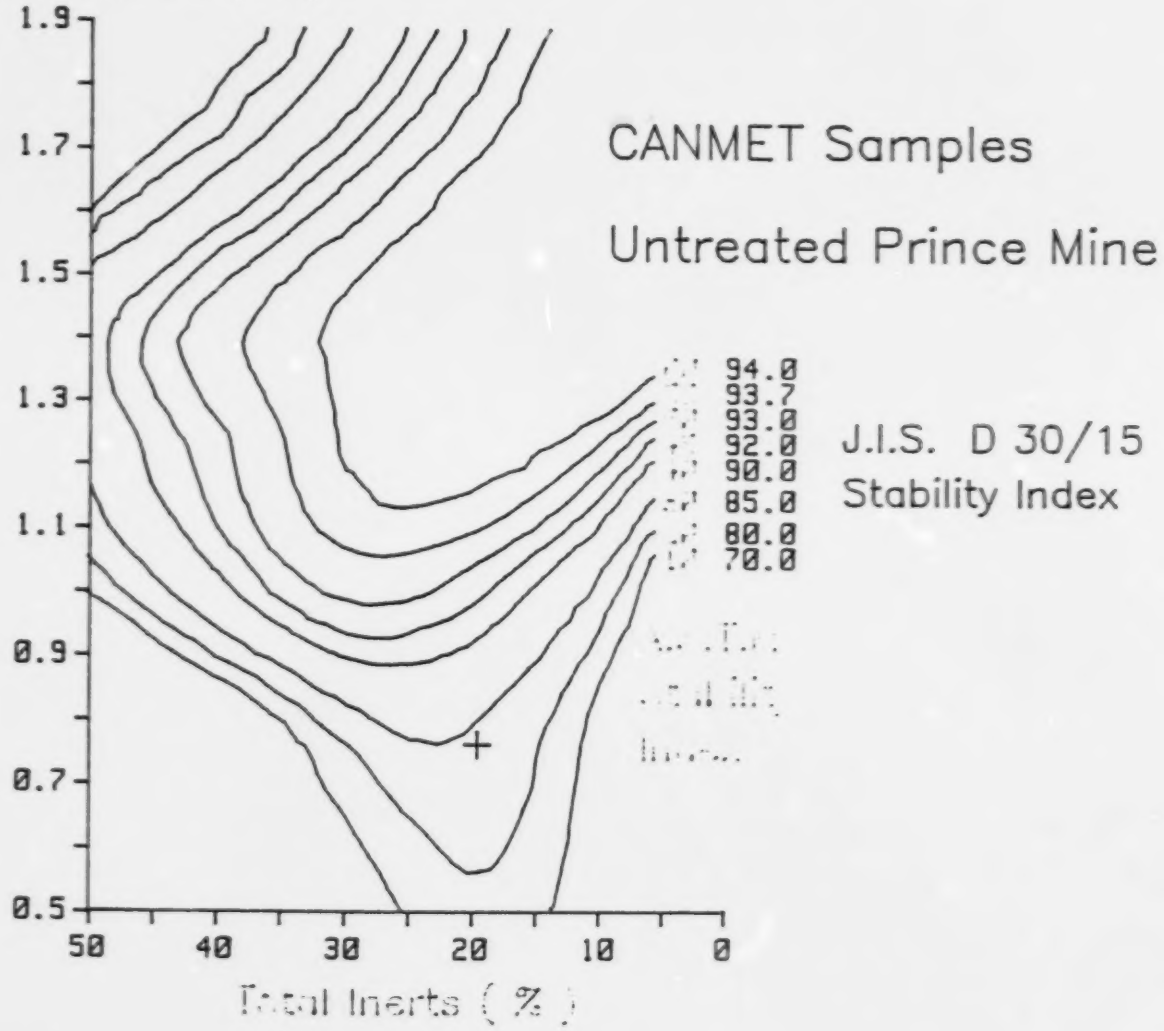
Vitrinite Types	%	Prorated Reactives	Optimum Ratio	PR/OR	Strength Factors	PRxSF
V-4	0	0.0	4.3	0.0	2.41	0.0
V-5	0	0.0	3.8	0.0	2.53	0.0
V-6	7	5.6	3.5	1.6	2.68	15.1
V-7	78	62.8	3.1	20.3	2.81	176.4
V-8	14	11.3	2.8	4.0	2.94	33.1
V-9	1	0.8	2.6	0.3	3.54	2.8
V-10	0	0.0	2.4	0.0	3.76	0.0
V-11	0	0.0	2.7	0.0	4.52	0.0
V-12	0	0.0	3.2	0.0	4.76	0.0
V-13	0	0.0	4.0	0.0	5.93	0.0
V-14	0	0.0	5.2	0.0	6.99	0.0
V-15	0	0.0	7.0	0.0	7.18	0.0
V-16	0	0.0	9.5	0.0	7.32	0.0
V-17	0	0.0	12.3	0.0	7.48	0.0
V-18	0	0.0	14.8	0.0	7.60	0.0
V-19	0	0.0	17.1	0.0	7.77	0.0
V-20	0	0.0	19.3	0.0	6.38	0.0

Optimum Inert Index = 26.2
Optimum Strength Index = 227.5

Composition Balance Index = 0.74
Strength Index = 2.83

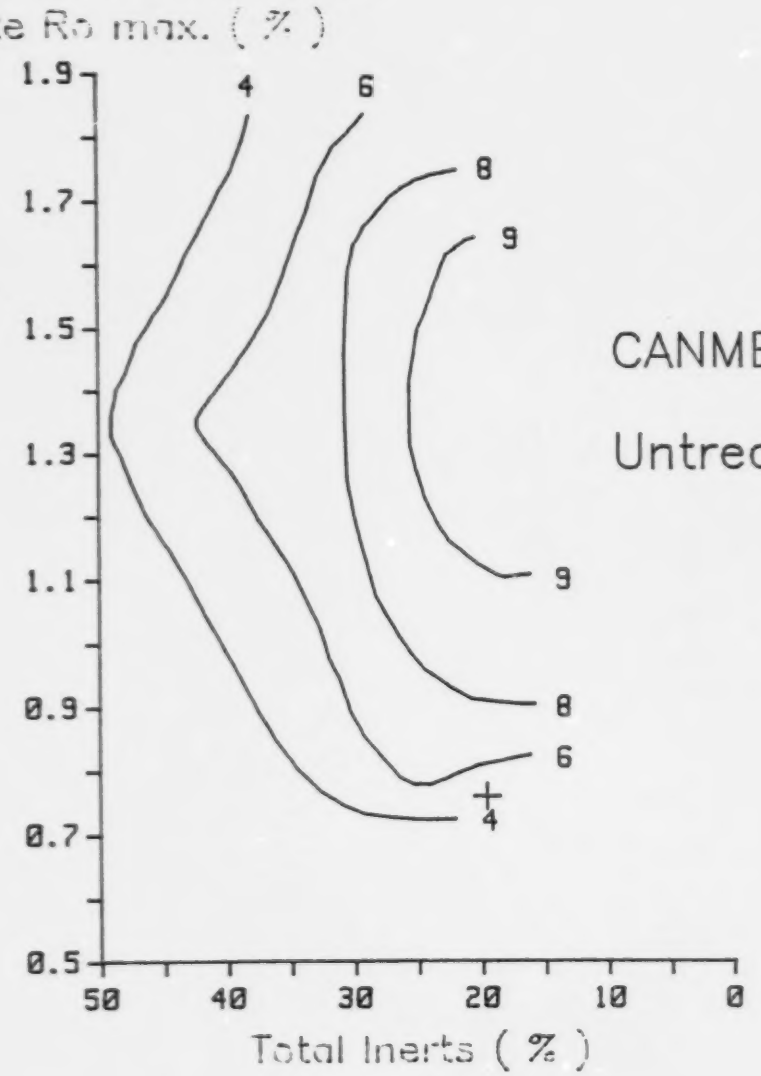
A.S.T.M. Stability Index = 29
J.I.S. D 30/15 = 85.0

PREDICTED COKE STRENGTH



David E. Pearson & Associates Ltd.

PREDICTED F.S.I.



CANMET Samples

Untreated Prince Mine

David E. Pearson & Associates Ltd.

Vitrinite Reflectance

CANMET SAMPLES
Untreated Lingan Mine Sample

BASIC STATISTICS

Total Number of Observations	100
Mean Maximum Reflectance (Romax)...%	.98
Standard Error of the Mean	.01
Coefficient of Variation...%	5.46
Variance	.0029
Standard Deviation	.0537
Skewness	-.3821
Kurtosis	3.2805

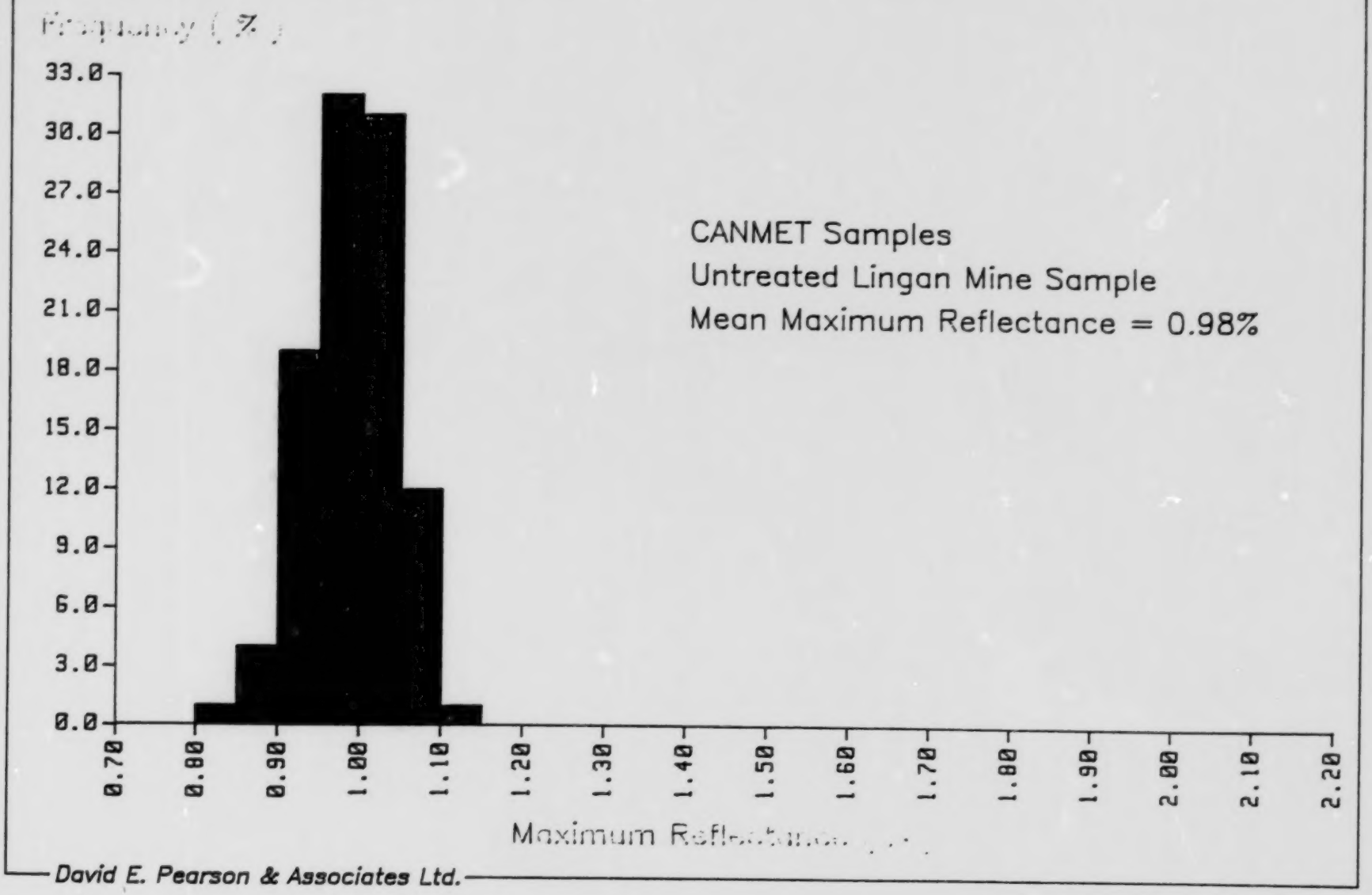
CELL STATISTICS

Cell Number	Lower Limit	Number of Observations	Frequency (%)
3	0.80	1	1.00
4	0.85	4	4.00
5	0.90	19	19.00
6	0.95	32	32.00
7	1.00	31	31.00
8	1.05	12	12.00
9	1.10	1	1.00

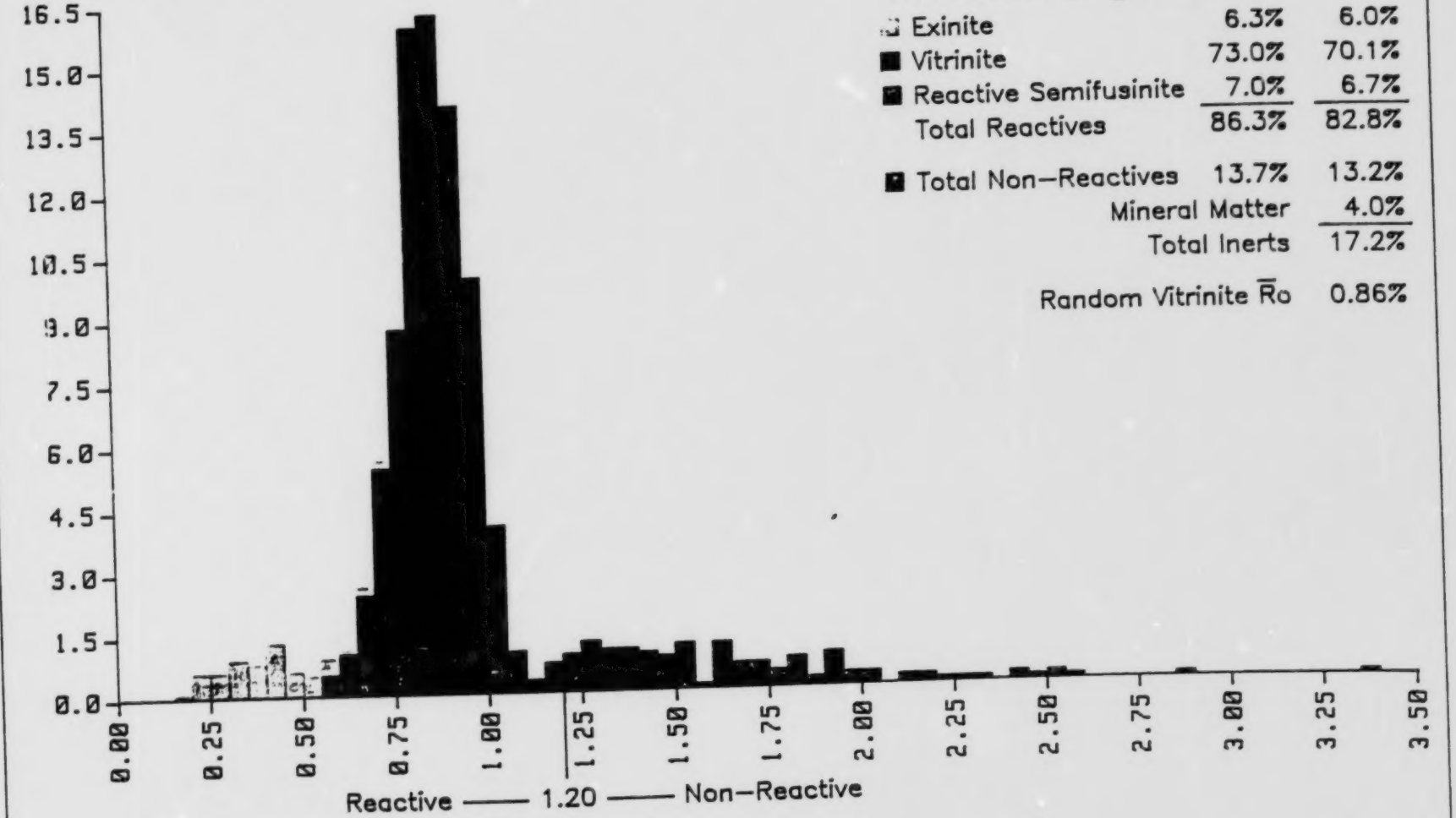
VITRINITE TYPE DISTRIBUTION

Vitrinite Type	Frequency (%)
V8	5.00
V9	51.00
V10	43.00
V11	1.00

VITRINITE HISTOGRAM



REFLECTOGRAM



CANMET Samples

Untreated Lingan Mine

Exinite	6.3%	6.0%
Vitrinite	73.0%	70.1%
Reactive Semifusinite	7.0%	6.7%
Total Reactives	86.3%	82.8%
Total Non—Reactives	13.7%	13.2%
Mineral Matter		4.0%
Total Inerts		17.2%
Random Vitrinite $\bar{R}o$		0.86%

David E. Pearson & Associates Ltd.

Maceral Analysis

CANMET SAMPLES
Untreated Lingan Mine

Count #	1	2	3	4	5	6	7	8	9	10
Vitrinite	74	71	71	77	71	81	71	73	64	77
Exinite	1	11	4	7	5	4	7	7	9	8
Reactive Semifusinite	9	9	11	1	13	3	6	10	4	4
Total Reactives	84	91	86	85	89	88	84	90	77	89
Macrinite	4	2	4	5	3	5	5	2	5	5
Inert Semifusinite	9	7	10	7	6	6	8	7	18	6
Fusinite	3	0	0	3	0	1	2	0	0	0
Inertodetrinite	0	0	0	0	2	0	1	1	0	0
Total Non-Reactives	16	9	14	15	11	12	16	10	23	11

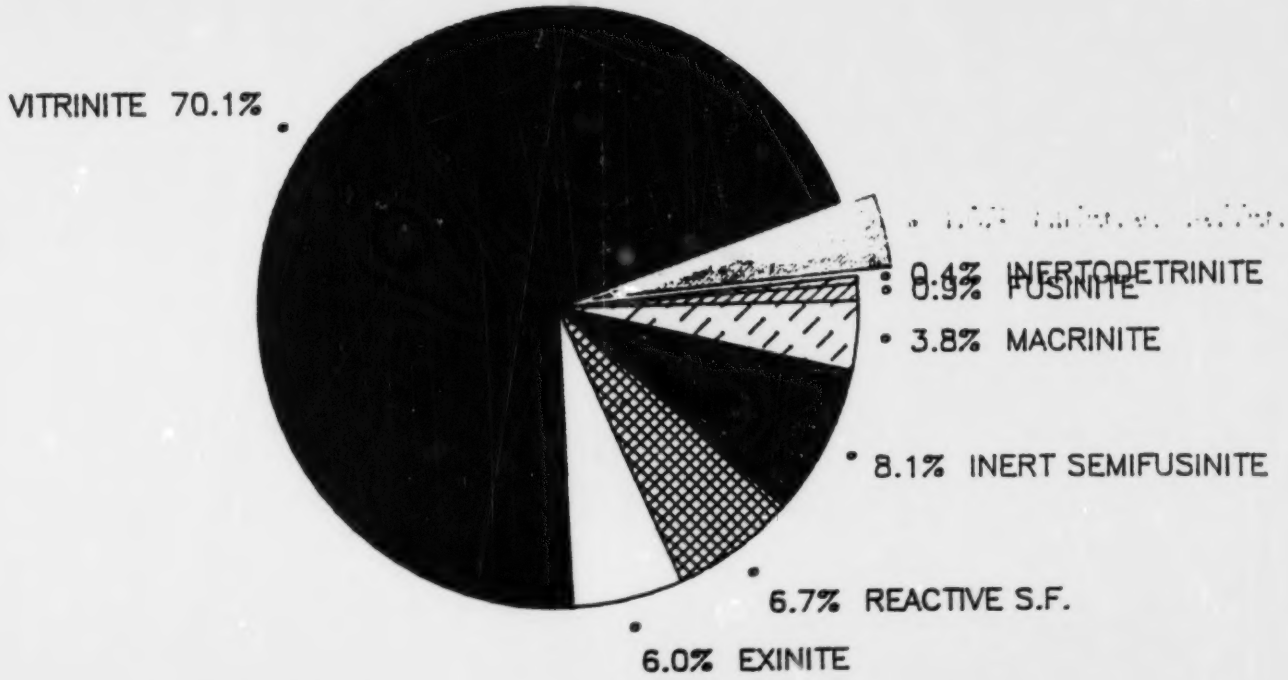
Basic Statistics	Mean	St.Deviation	Variance
Vitrinite	73.0	4.6	21.6
Exinite	6.3	2.9	8.2
Reactive Semifusinite	7.0	3.9	15.6
Total Reactives	86.3	4.1	16.9
Macrinite	4.0	1.3	1.6
Inert Semifusinite	8.4	3.6	13.2
Fusinite	.9	1.3	1.7
Inertodetrinite	.4	.7	.5
Total Non-Reactives	13.7	4.1	16.9

Maceral Data Corrected For Mineral Matter Content

Vitrinite	70.1
Exinite	6.0
Reactive Semifusinite	6.7
Total Reactives	82.8
Macrinite	3.8
Inert Semifusinite	8.1
Fusinite	.9
Inertodetrinite	.4
Total Non-Reactives	13.2
Mineral Matter	4.0
Total Inerts	17.2

MACERAL DISTRIBUTION

Semifusinite – Pearson Method



CANMET Samples
Untreated Lingan Mine

David E. Pearson & Associates Ltd.

Coke Stability Calculation

CANMET SAMPLES
Untreated Lingan Mine

Coke Stability Index Calculation

Total Inerts = 17.2 % (100 - TI) / 100 = 0.83

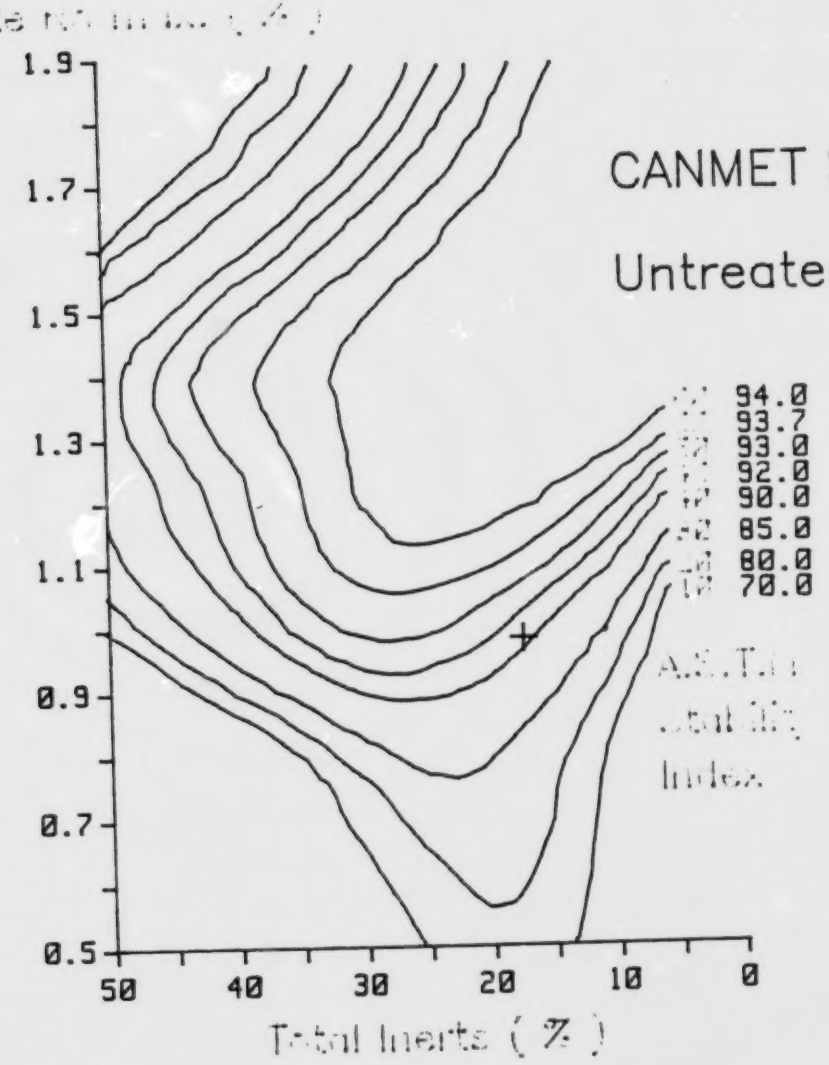
Vitrinite Types	%	Prorated Reactives	Optimum Ratio	PR/OR	Strength Factors	PRxSF
V-4	0	0.0	4.3	0.0	2.43	0.0
V-5	0	0.0	3.8	0.0	2.55	0.0
V-6	0	0.0	3.5	0.0	2.66	0.0
V-7	0	0.0	3.1	0.0	2.79	0.0
V-8	5	4.1	2.8	1.5	2.89	12.0
V-9	51	42.2	2.6	16.2	3.48	147.0
V-10	43	35.6	2.4	14.8	3.70	131.7
V-11	1	0.8	2.7	0.3	4.45	3.7
V-12	0	0.0	3.2	0.0	4.71	0.0
V-13	0	0.0	4.0	0.0	5.91	0.0
V-14	0	0.0	5.2	0.0	7.03	0.0
V-15	0	0.0	7.0	0.0	7.20	0.0
V-16	0	0.0	9.5	0.0	7.35	0.0
V-17	0	0.0	12.3	0.0	7.52	0.0
V-18	0	0.0	14.8	0.0	7.63	0.0
V-19	0	0.0	17.1	0.0	7.79	0.0
V-20	0	0.0	19.3	0.0	6.50	0.0

Optimum Inert Index = 32.9
Optimum Strength Index = 294.3

Composition Balance Index = 0.52
Strength Index = 3.55

A.S.T.M. Stability Index = 42
J.I.S. D 30/15 = 91.0

PREDICTED COKE STRENGTH



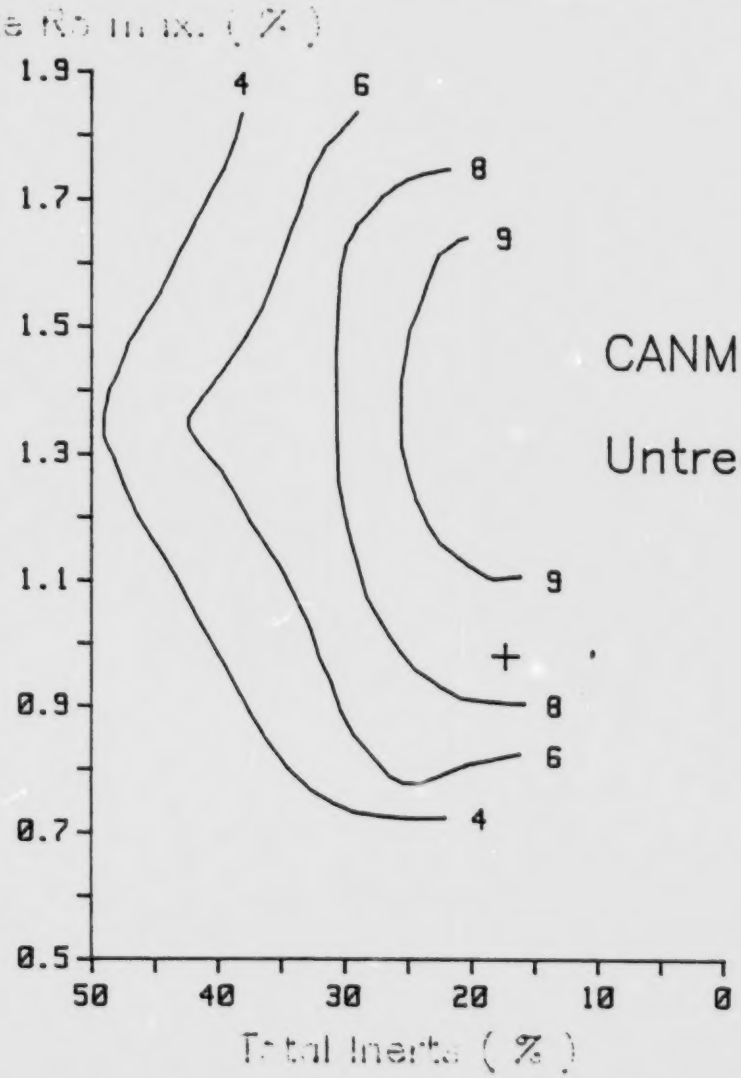
CANMET Samples

Untreated Lingan Mine

J.I.S. D 30/15
Stability Index

David E. Pearson & Associates Ltd.

PREDICTED F.S.I.

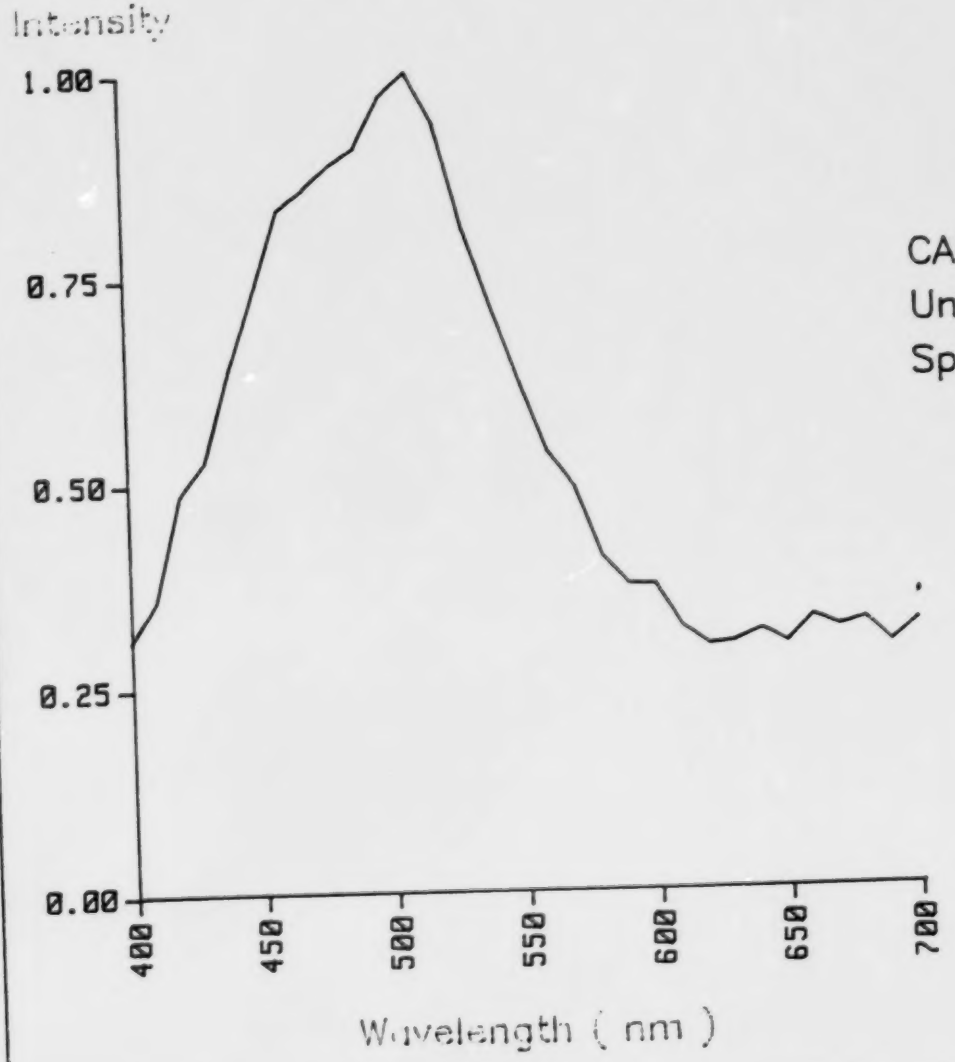


CANMET Samples

Untreated Lingan Mine

David E. Pearson & Associates Ltd.

FLUORESCENCE SPECTRUM



CANMET Samples
Untreated Prince Mine Sample
Sporinite Maceral

$$Q \frac{\text{Intensity @ 650 nm}}{\text{Intensity @ 500 nm}} = 0.30$$

Maximum occurs at 510 nm

David E. Pearson & Associates Ltd.

Vitrinite Reflectance

CANMET SAMPLES
DSS Sample #1

BASIC STATISTICS

Total Number of Observations.....	100
Mean Maximum Reflectance (Romax)...%	.53
Standard Error of the Mean.....	.01
Coefficient of Variation.....%	9.66
Variance.....	.0026
Standard Deviation.....	.0510
Skewness.....	.4110
Kurtosis.....	2.4962

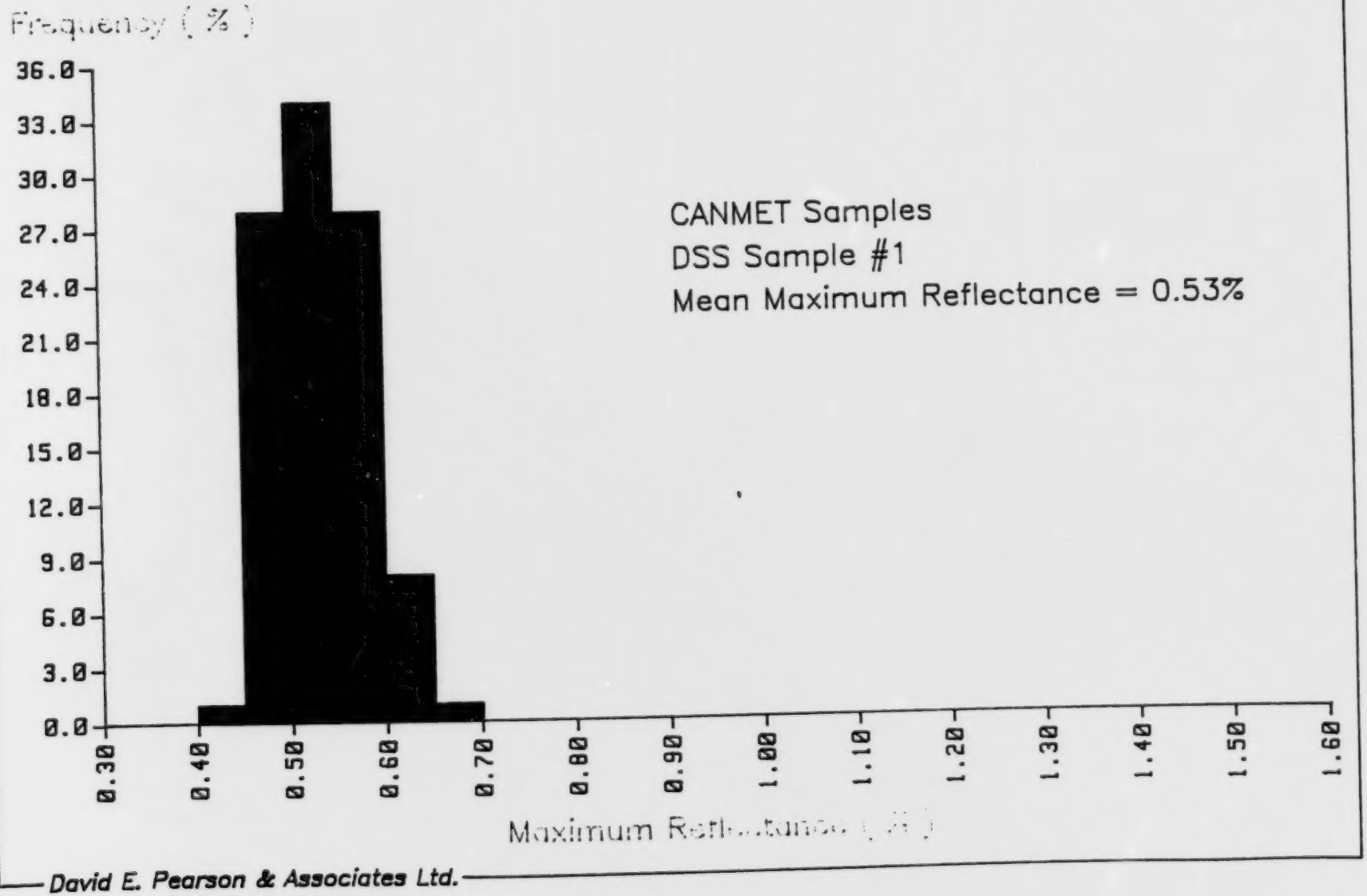
CELL STATISTICS

Cell Number	Lower Limit	Number of Observations	Frequency (%)
3	0.40	1	1.00
4	0.45	28	28.00
5	0.50	34	34.00
6	0.55	28	28.00
7	0.60	8	8.00
8	0.65	1	1.00

VITRINITE TYPE DISTRIBUTION

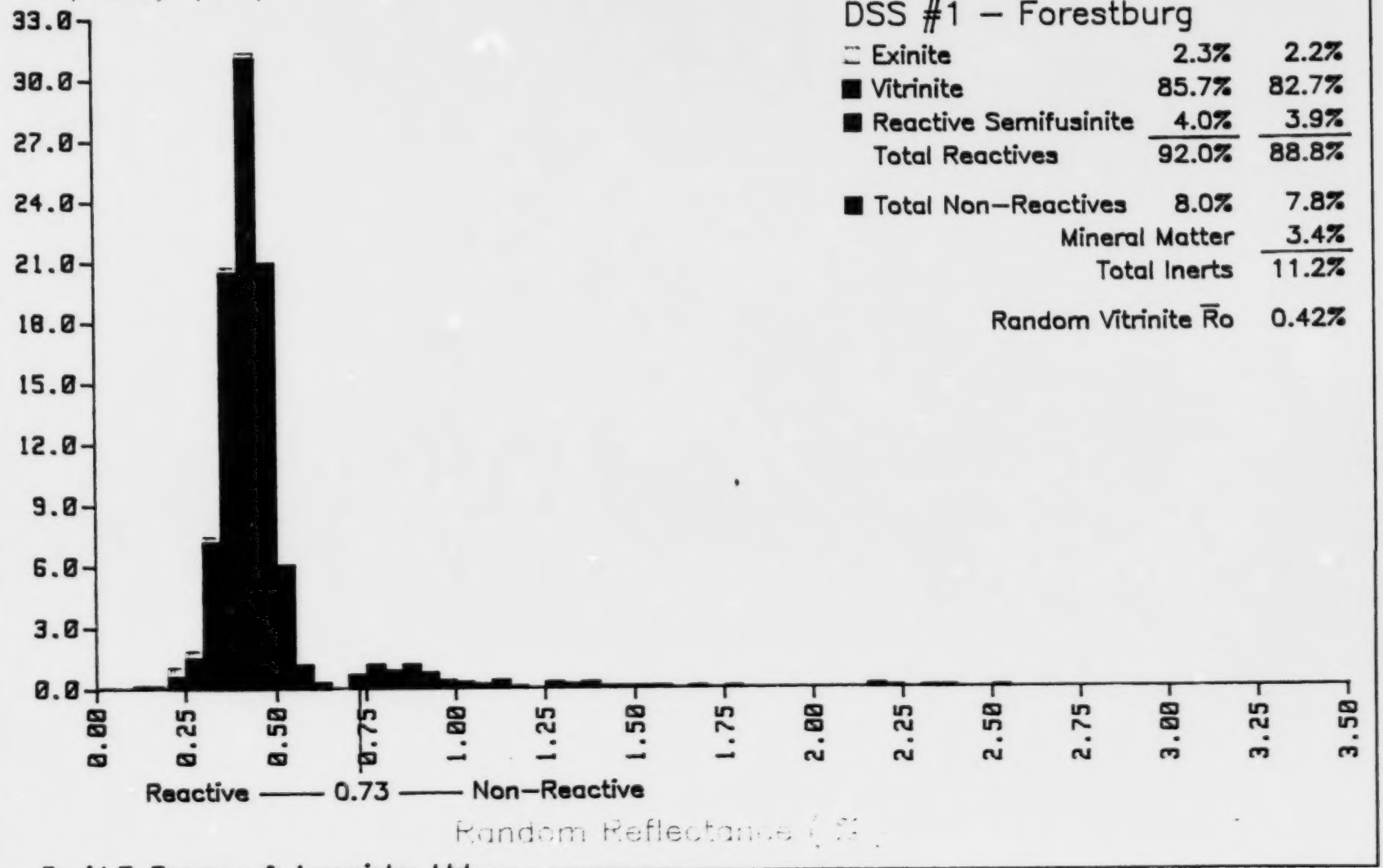
Vitrinite Type	Frequency (%)
V4	29.00
V5	62.00
V6	9.00

VITRINITE HISTOGRAM



David E. Pearson & Associates Ltd.

REFLECTOGRAM



CANMET Samples

DSS #1 – Forestburg

Exinite	2.3%	2.2%
Vitrinite	85.7%	82.7%
Reactive Semifusinite	4.0%	3.9%
Total Reactives	92.0%	88.8%
Total Non—Reactives	8.0%	7.8%
Mineral Matter		3.4%
Total Inerts		11.2%
Random Vitrinite $\overline{R}o$		0.42%

David E. Pearson & Associates Ltd.

Maceral Analysis

CANMET SAMPLES
DSS #1 - Forestburg

Count #	1	2	3	4	5	6	7	8	9	10
Vitrinite	95	88	85	85	86	93	83	85	79	78
Exinite	1	2	2	1	1	1	2	2	7	4
Reactive Semifusinite	2	2	7	5	3	1	5	3	7	5
Total Reactives	98	92	94	91	90	95	90	90	93	87
Macrinite	0	3	3	6	3	1	2	2	1	6
Inert Semifusinite	0	0	1	3	2	3	1	3	0	0
Fusinite	0	0	1	0	0	0	2	3	5	2
Inertodetrinite	2	5	1	0	5	1	5	2	1	5
Total Non-Reactives	2	8	6	9	10	5	10	10	7	13

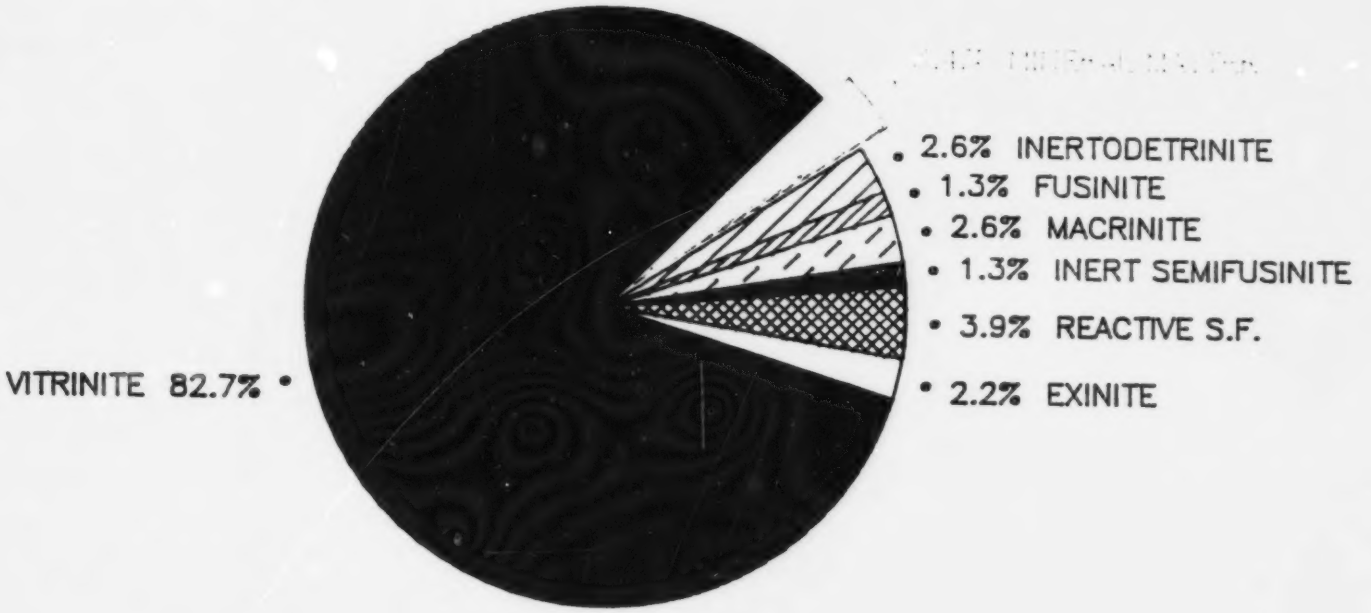
Basic Statistics	Mean	St.Deviation	Variance
Vitrinite	85.7	5.4	28.7
Exinite	2.3	1.9	3.6
Reactive Semifusinite	4.0	2.1	4.4
Total Reactives	92.0	3.1	9.8
Macrinite	2.7	2.0	4.0
Inert Semifusinite	1.3	1.3	1.8
Fusinite	1.3	1.7	2.9
Inertodetrinite	2.7	2.0	4.2
Total Non-Reactives	8.0	3.1	9.8

Maceral Data Corrected For Mineral Matter Content

Vitrinite	82.7
Exinite	2.2
Reactive Semifusinite	3.9
Total Reactives	88.8
Macrinite	2.6
Inert Semifusinite	1.3
Fusinite	1.3
Inertodetrinite	2.6
Total Non-Reactives	7.8
Mineral Matter	3.4
Total Inerts	11.2

MACERAL DISTRIBUTION

Semifusinite – Pearson Method



CANMET Samples
DSS #1 – Forestburg

David E. Pearson & Associates Ltd.

Vitrinite Reflectance

CANMET SAMPLES
DSS Sample #2

BASIC STATISTICS

Total Number of Observations	100
Mean Maximum Reflectance (Romax)...%	.55
Standard Error of the Mean	0.00
Coefficient of Variation....%	9.06
Variance	.0025
Standard Deviation	.0495
Skewness	.8179
Kurtosis	4.3543

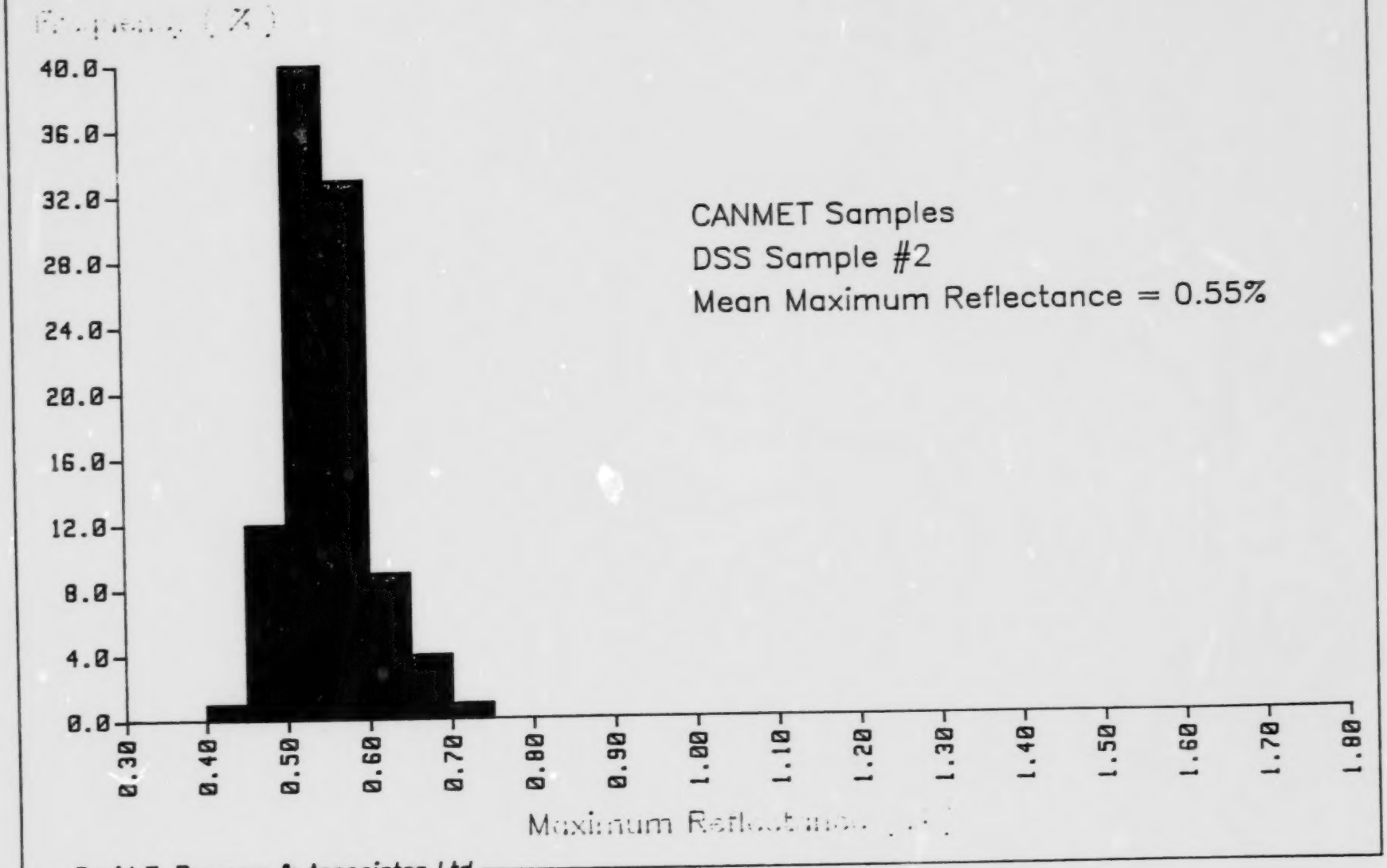
CELL STATISTICS

Cell Number	Lower Limit	Number of Observations	Frequency (%)
3	0.40	1	1.00
4	0.45	12	12.00
5	0.50	40	40.00
6	0.55	33	33.00
7	0.60	9	9.00
8	0.65	4	4.00
9	0.70	1	1.00

VITRINITE TYPE DISTRIBUTION

Vitrinite Type	Frequency (%)
V4	13.00
V5	73.00
V6	13.00
V7	1.00

VITRINITE HISTOGRAM



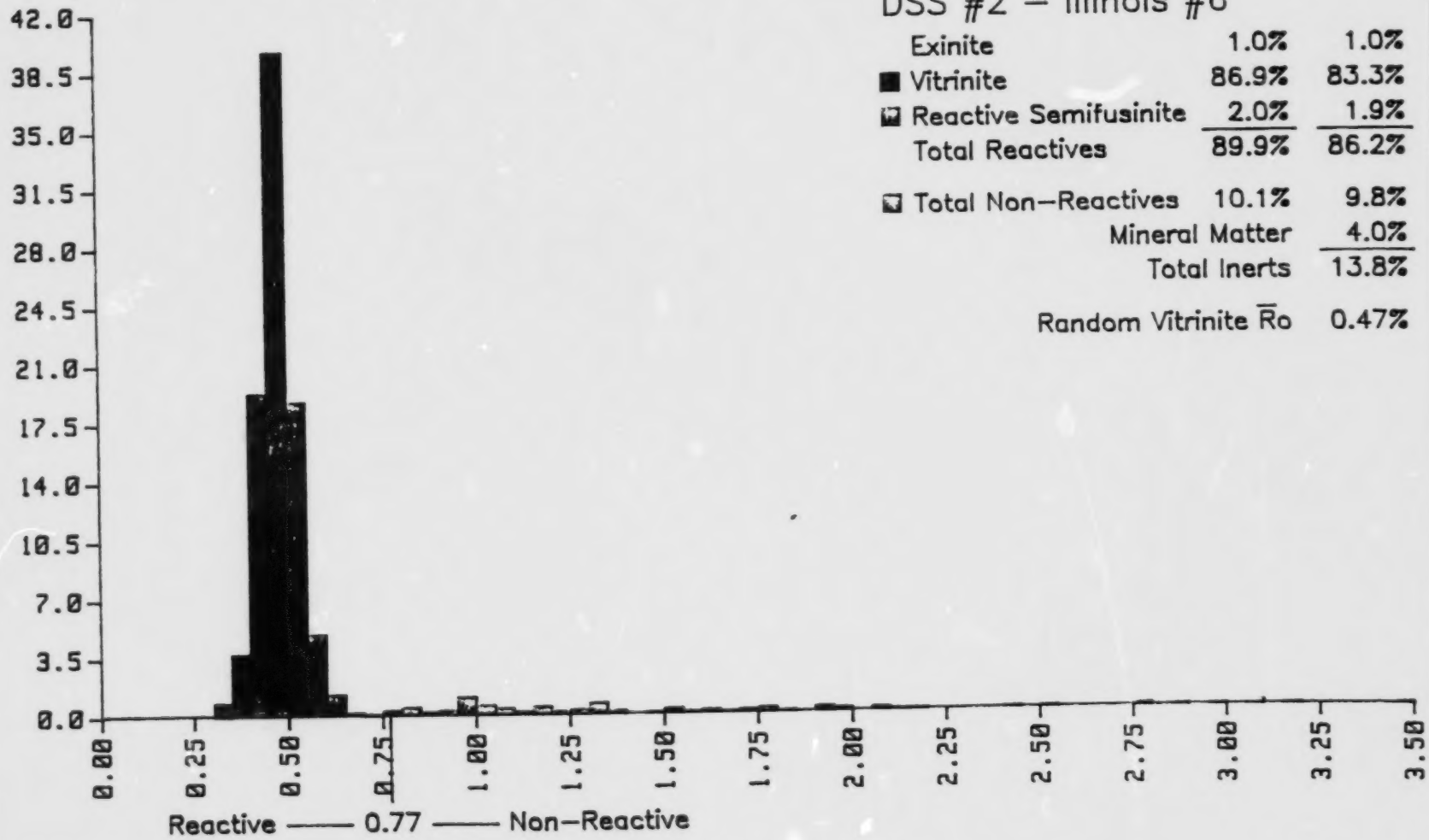
David E. Pearson & Associates Ltd.

REFLECTOGRAM

CANMET Samples

DSS #2 – Illinois #6

Exinite	1.0%	1.0%
■ Vitrinite	86.9%	83.3%
▣ Reactive Semifusinite	2.0%	1.9%
Total Reactives	89.9%	86.2%
▣ Total Non–Reactives	10.1%	9.8%
Mineral Matter		4.0%
Total Inerts		13.8%
Random Vitrinite $\overline{R}o$		0.47%



David E. Pearson & Associates Ltd.

Maceral Analysis

CANMET SAMPLES
DSS #2 - Illinois #6

Count #	1	2	3	4	5	6	7	8	9	10
Vitrinite	85	88	89	89	87	85	85	90	83	88
Exinite	2	3	1	0	0	0	2	0	1	1
Reactive Semifusinite	3	2	0	2	2	2	6	1	2	0
Total Reactives	90	93	90	91	89	87	93	91	86	89
Macrinite	4	3	5	4	4	9	4	5	7	6
Inert Semifusinite	4	2	3	3	5	4	2	4	3	1
Fusinite	1	2	1	1	1	0	0	0	3	1
Inertodetrinite	1	0	1	1	1	0	1	0	1	3
Total Non-Reactives	10	7	10	9	11	13	7	9	14	11

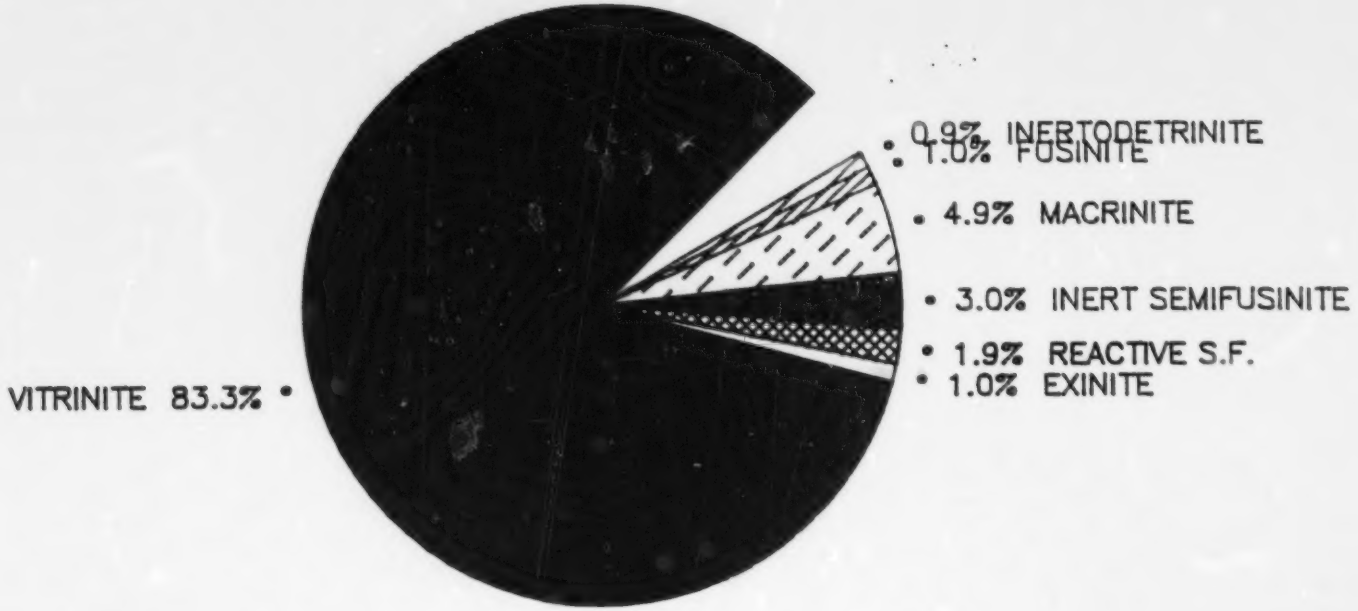
Basic Statistics	Mean	St.Deviation	Variance
Vitrinite	86.9	2.3	5.2
Exinite	1.0	1.0	1.1
Reactive Semifusinite	2.0	1.7	2.9
Total Reactives	89.9	2.3	5.2
Macrinite	5.1	1.8	3.2
Inert Semifusinite	3.1	1.2	1.4
Fusinite	1.0	.9	.9
Inertodetrinite	.9	.9	.8
Total Non-Reactives	10.1	2.3	5.2

Maceral Data Corrected For Mineral Matter Content

Vitrinite	83.3
Exinite	1.0
Reactive Semifusinite	1.9
Total Reactives	86.2
Macrinite	4.9
Inert Semifusinite	3.0
Fusinite	1.0
Inertodetrinite	.9
Total Non-Reactives	9.8
Mineral Matter	4.0
Total Inerts	13.8

MACERAL DISTRIBUTION

Semifusinite – Pearson Method



CANMET Samples
DSS #2 – Illinois #6

David E. Pearson & Associates Ltd.

Coke Stability Calculation

CANMET SAMPLES
DSS #2 - Illinois #6

Coke Stability Index Calculation

Total Inerts = 13.8 %

(100 - TI) / 100 = 0.86

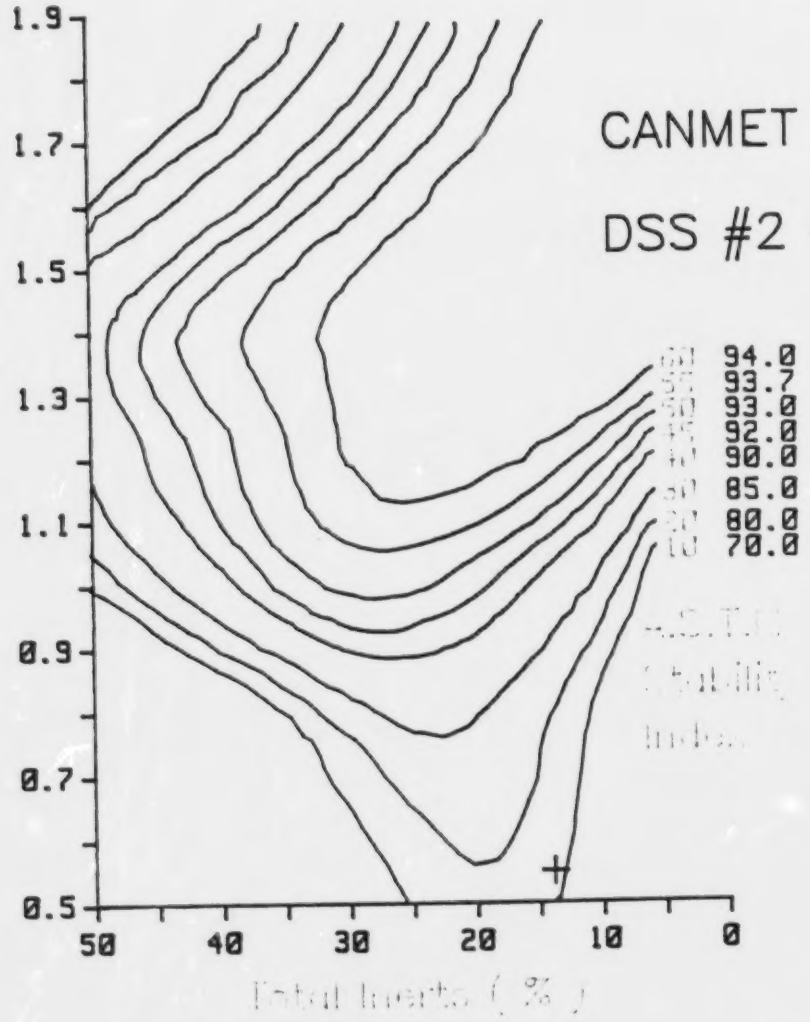
Vitrinite Types	%	Prorated Reactives	Optimum Ratio	PR/OR	Strength Factors	PRxSF
V-4	13	11.2	4.3	2.6	2.41	27.0
V-5	73	62.9	3.8	16.6	2.52	158.6
V-6	13	11.2	3.5	3.2	2.61	29.2
V-7	1	0.9	3.1	0.3	2.73	2.4
V-8	0	0.0	2.8	0.0	2.83	0.0
V-9	0	0.0	2.6	0.0	3.36	0.0
V-10	0	0.0	2.4	0.0	3.60	0.0
V-11	0	0.0	2.7	0.0	4.36	0.0
V-12	0	0.0	3.2	0.0	4.62	0.0
V-13	0	0.0	4.0	0.0	5.85	0.0
V-14	0	0.0	5.2	0.0	7.04	0.0
V-15	0	0.0	7.0	0.0	7.23	0.0
V-16	0	0.0	9.5	0.0	7.38	0.0
V-17	0	0.0	12.3	0.0	7.54	0.0
V-18	0	0.0	14.8	0.0	7.67	0.0
V-19	0	0.0	17.1	0.0	7.81	0.0
V-20	0	0.0	19.3	0.0	6.61	0.0

Optimum Inert Index = 22.6
Optimum Strength Index = 217.2

Composition Balance Index = 0.61
Strength Index = 2.52

A.S.T.M. Stability Index = 13
J.I.S. D 30/15 = 73.3

PREDICTED COKE STRENGTH



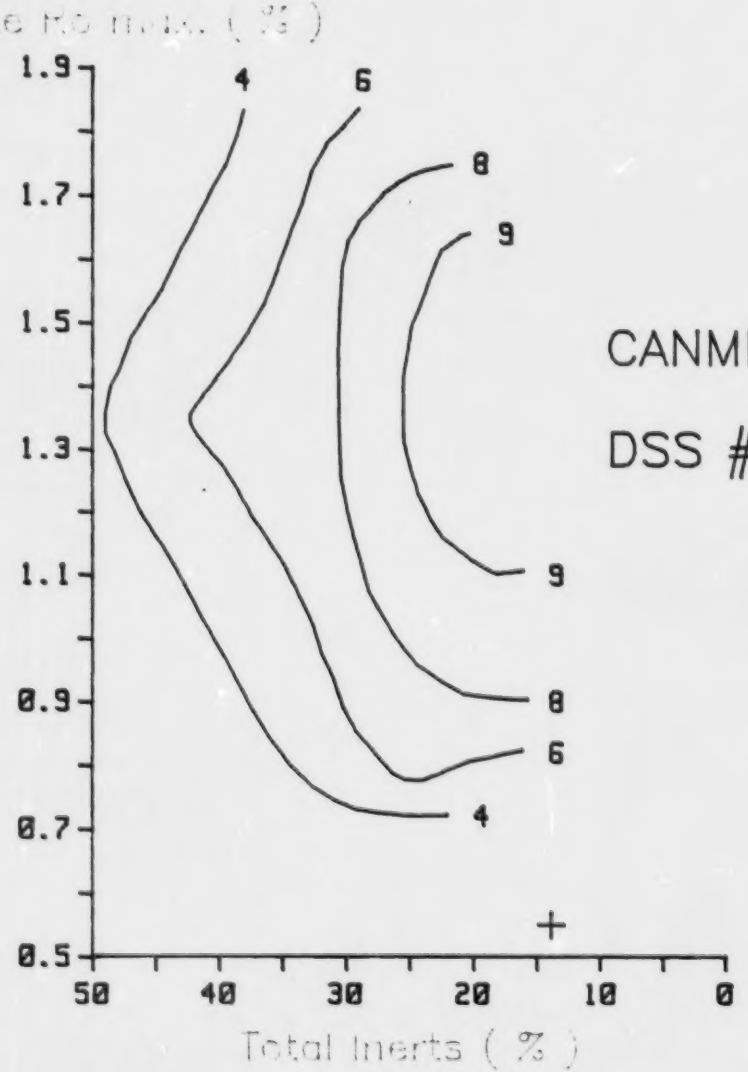
CANMET Samples

DSS #2 – Illinois #6

J.I.S. D 30/15
Stability Index

David E. Pearson & Associates Ltd.

PREDICTED F.S.I.



CANMET Samples

DSS #2 – Illinois #6

David E. Pearson & Associates Ltd.

Vitrinite Reflectance

CANMET SAMPLES
DDS Sample #3

BASIC STATISTICS

Total Number of Observations	100
Mean Maximum Reflectance (Romax)...%	.72
Standard Error of the Mean	0.00
Coefficient of Variation...%	6.23
Variance	.0020
Standard Deviation	.0447
Skewness	.7835
Kurtosis	3.5914

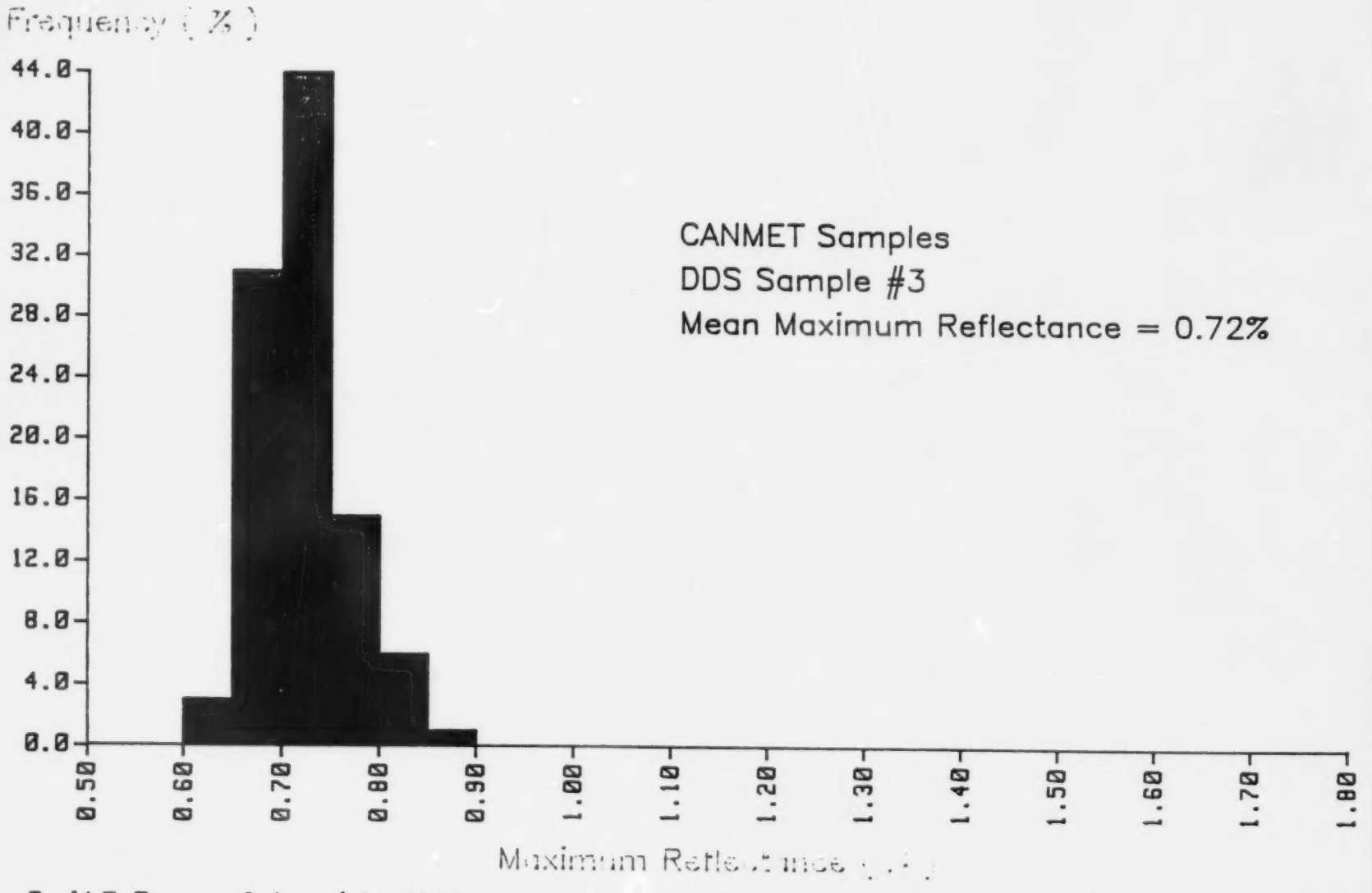
CELL STATISTICS

Cell Number	Lower Limit	Number of Observations	Frequency (%)
3	0.60	3	3.00
4	0.65	31	31.00
5	0.70	44	44.00
6	0.75	15	15.00
7	0.80	6	6.00
8	0.85	1	1.00

VITRINITE TYPE DISTRIBUTION

Vitrinite Type	Frequency (%)
V6	34.00
V7	59.00
V8	7.00

VITRINITE HISTOGRAM



David E. Pearson & Associates Ltd.

Vitrinite Reflectance

CANMET SAMPLES
DSS Sample #4

BASIC STATISTICS

Total Number of Observations	100
Mean Maximum Reflectance (Romax)...%	.91
Standard Error of the Mean	.01
Coefficient of Variation...%	6.17
Variance	.0032
Standard Deviation	.0562
Skewness	-.3707
Kurtosis	3.1046

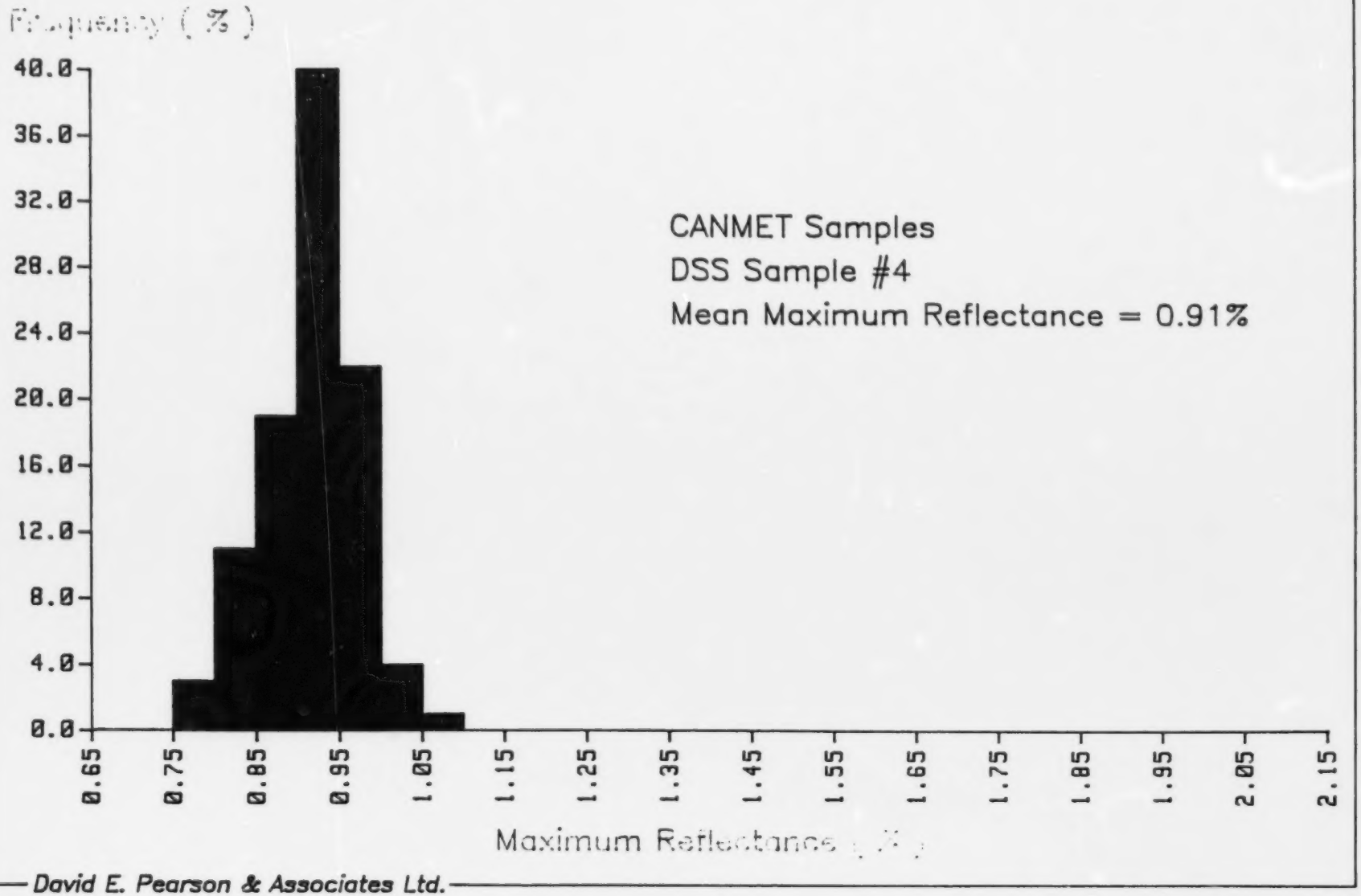
CELL STATISTICS

Cell Number	Lower Limit	Number of Observations	Frequency (%)
3	0.75	3	3.00
4	0.80	11	11.00
5	0.85	19	19.00
6	0.90	40	40.00
7	0.95	22	22.00
8	1.00	4	4.00
9	1.05	1	1.00

VITRINITE TYPE DISTRIBUTION

Vitrinite Type	Frequency (%)
V7	3.00
V8	30.00
V9	62.00
V10	5.00

VITRINITE HISTOGRAM



David E. Pearson & Associates Ltd.

Vitrinite Reflectance

CANMET SAMPLES
Sample DSS #5

BASIC STATISTICS

Total Number of Observations	100
Mean Maximum Reflectance (Romax)...%	.63
Standard Error of the Mean	.01
Coefficient of Variation...%	9.40
Variance	.0035
Standard Deviation	.0592
Skewness	.6308
Kurtosis	3.3811

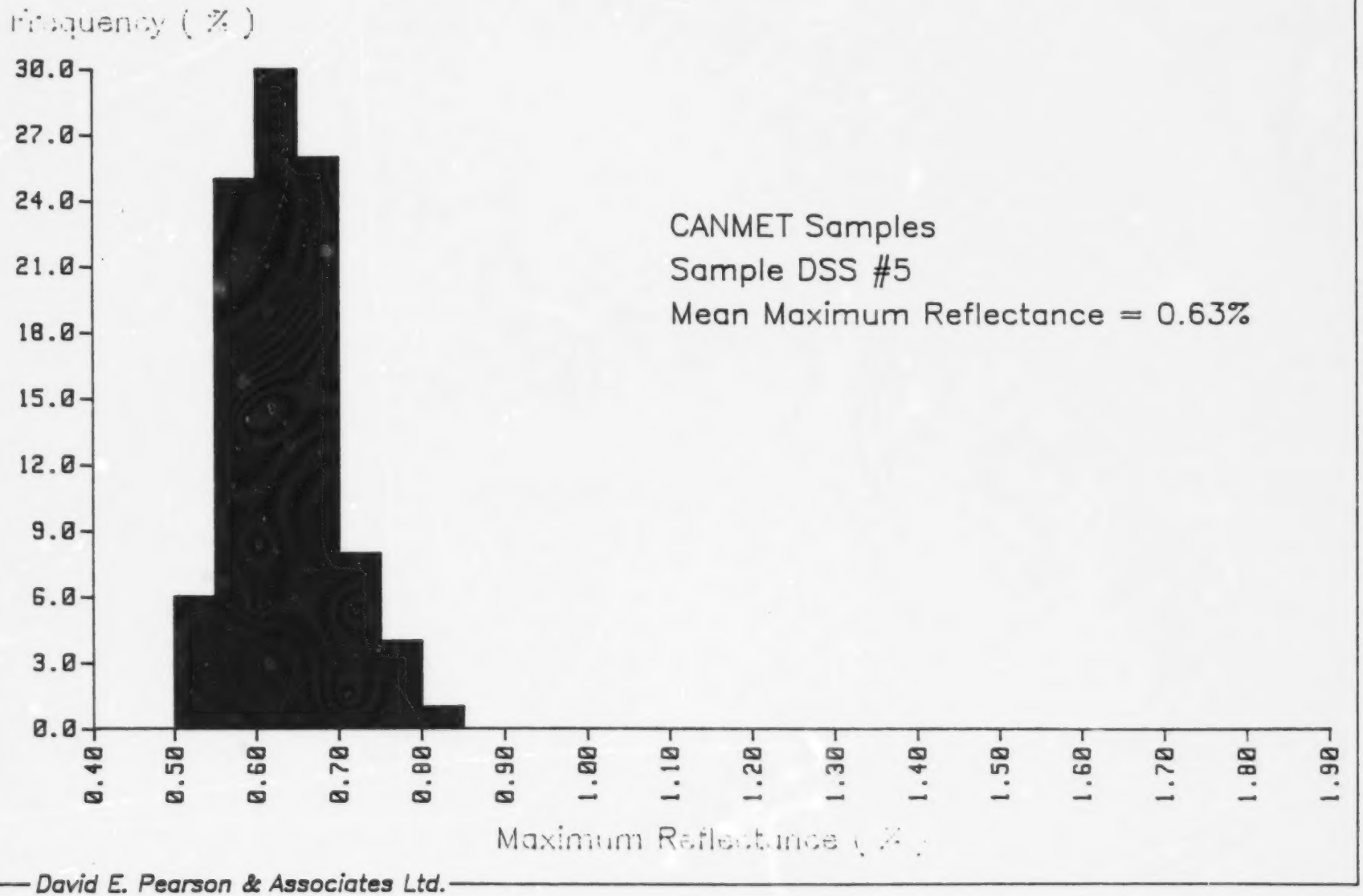
CELL STATISTICS

Cell Number	Lower Limit	Number of Observations	Frequency (%)
3	0.50	6	6.00
4	0.55	25	25.00
5	0.60	30	30.00
6	0.65	26	26.00
7	0.70	8	8.00
8	0.75	4	4.00
9	0.80	1	1.00

VITRINITE TYPE DISTRIBUTION

Vitrinite Type	Frequency (%)
V5	31.00
V6	56.00
V7	12.00
V8	1.00

VITRINITE HISTOGRAM



David E. Pearson & Associates Ltd.

Vitrinite Reflectance

CANMET SAMPLES
DDS Sample #6

BASIC STATISTICS

Total Number of Observations	100
Mean Maximum Reflectance (Romax)...%	.52
Standard Error of the Mean	0.00
Coefficient of Variation...%	8.13
Variance	.0018
Standard Deviation	.0424
Skewness	.8956
Kurtosis	3.9461

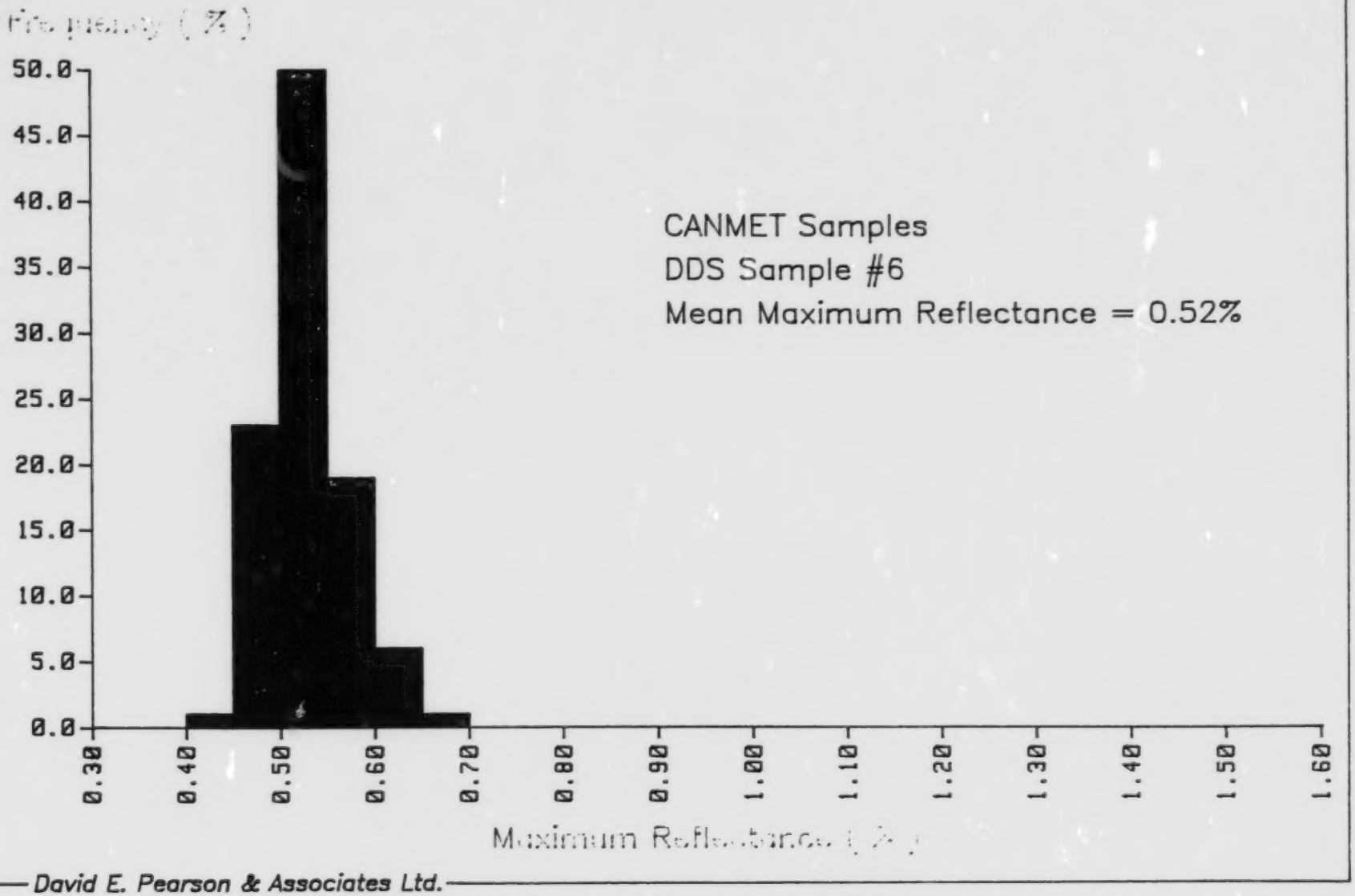
CELL STATISTICS

Cell Number	Lower Limit	Number of Observations	Frequency (%)
3	0.40	1	1.00
4	0.45	23	23.00
5	0.50	50	50.00
6	0.55	19	19.00
7	0.60	6	6.00
8	0.65	1	1.00

VITRINITE TYPE DISTRIBUTION

Vitrinite Type	Frequency (%)
V4	24.00
V5	69.00
V6	7.00

VITRINITE HISTOGRAM



David E. Pearson & Associates Ltd.

Vitrinite Reflectance

CANMET SAMPLES
DSS sAMPLE #7

BASIC STATISTICS

Total Number of Observations.....	100
Mean Maximum Reflectance (Romax)...%	.61
Standard Error of the Mean.....	0.00
Coefficient of Variation.....%	7.17
Variance.....	.0019
Standard Deviation.....	.0438
Skewness.....	.3163
Kurtosis.....	3.5630

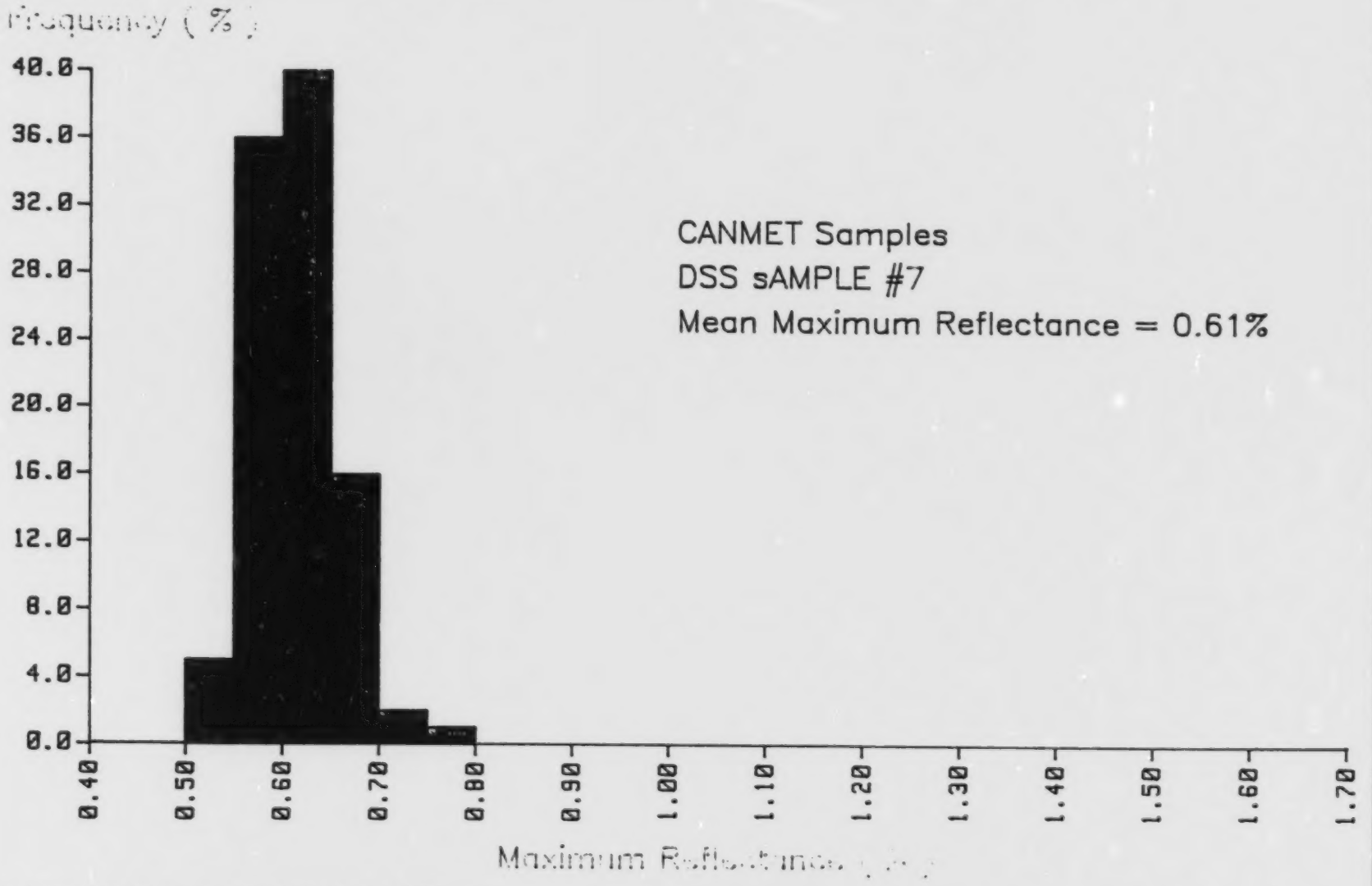
CELL STATISTICS

Cell Number	Lower Limit	Number of Observations	Frequency (%)
3	0.50	5	5.00
4	0.55	36	36.00
5	0.60	40	40.00
6	0.65	16	16.00
7	0.70	2	2.00
8	0.75	1	1.00

VITRINITE TYPE DISTRIBUTION

Vitrinite Type	Frequency (%)
V5	41.00
V6	56.00
V7	3.00

VITRINITE HISTOGRAM



David E. Pearson & Associates Ltd.

Vitrinite Reflectance

CANMET SAMPLES
DSS Sample #8

BASIC STATISTICS

Total Number of Observations	100
Mean Maximum Reflectance (Romax)...%	.97
Standard Error of the Mean	0.00
Coefficient of Variation...%	4.84
Variance	.0022
Standard Deviation	.0468
Skewness	-.1612
Kurtosis	2.8482

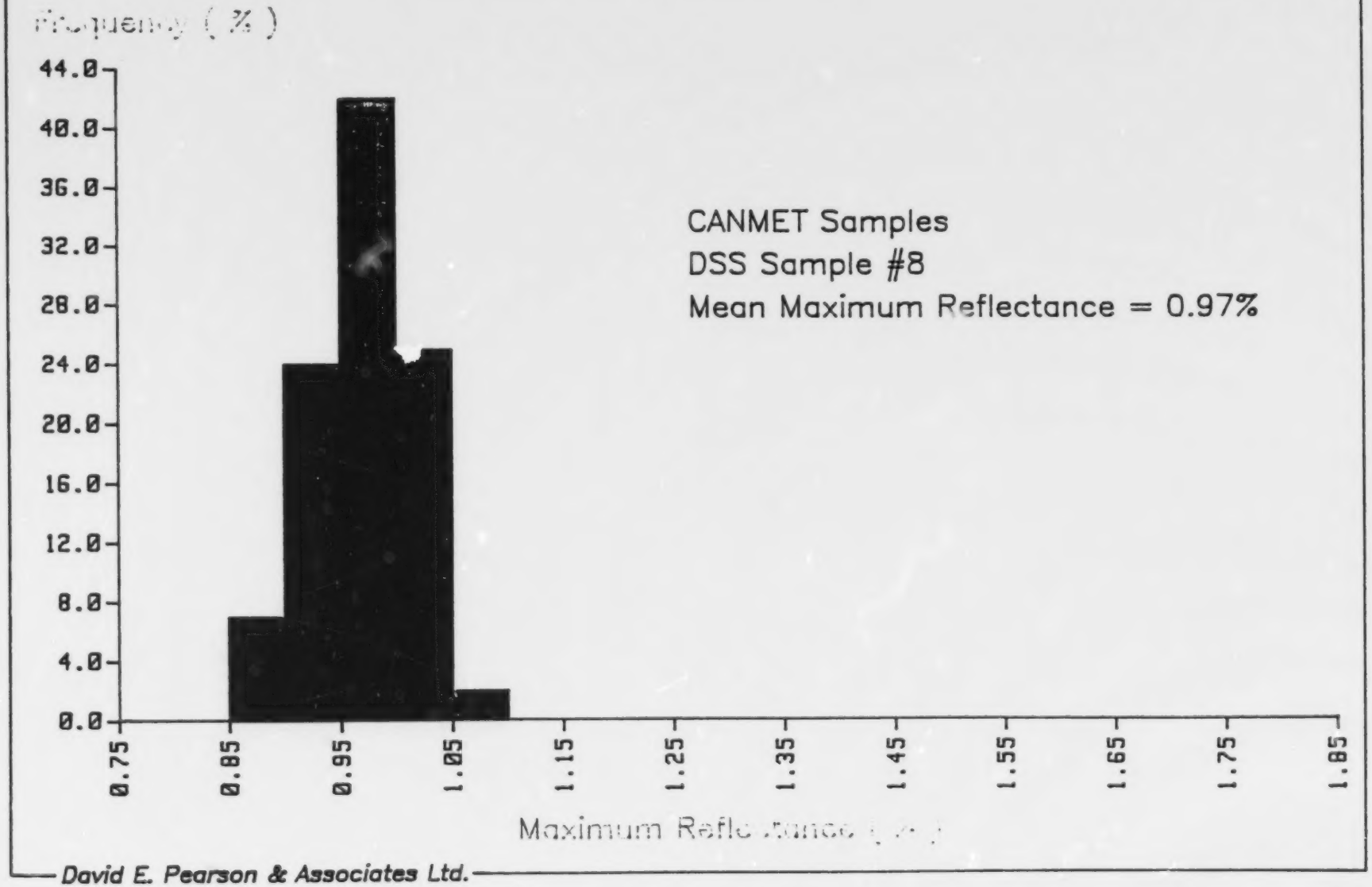
CELL STATISTICS

Cell Number	Lower Limit	Number of Observations	Frequency (%)
10	0.85	7	7.00
11	0.90	24	24.00
12	0.95	42	42.00
13	1.00	25	25.00
14	1.05	2	2.00

VITRINITE TYPE DISTRIBUTION

Vitrinite Type	Frequency (%)
V8	7.00
V9	66.00
V10	27.00

VITRINITE HISTOGRAM



David E. Pearson & Associates Ltd.

Vitrinite Reflectance

CANMET SAMPLES
DSS Sample #9

BASIC STATISTICS

Total Number of Observations	100
Mean Maximum Reflectance (Romax)...%	.55
Standard Error of the Mean	.01
Coefficient of Variation...%	9.18
Variance	.0026
Standard Deviation	.0505
Skewness	.4198
Kurtosis	3.5435

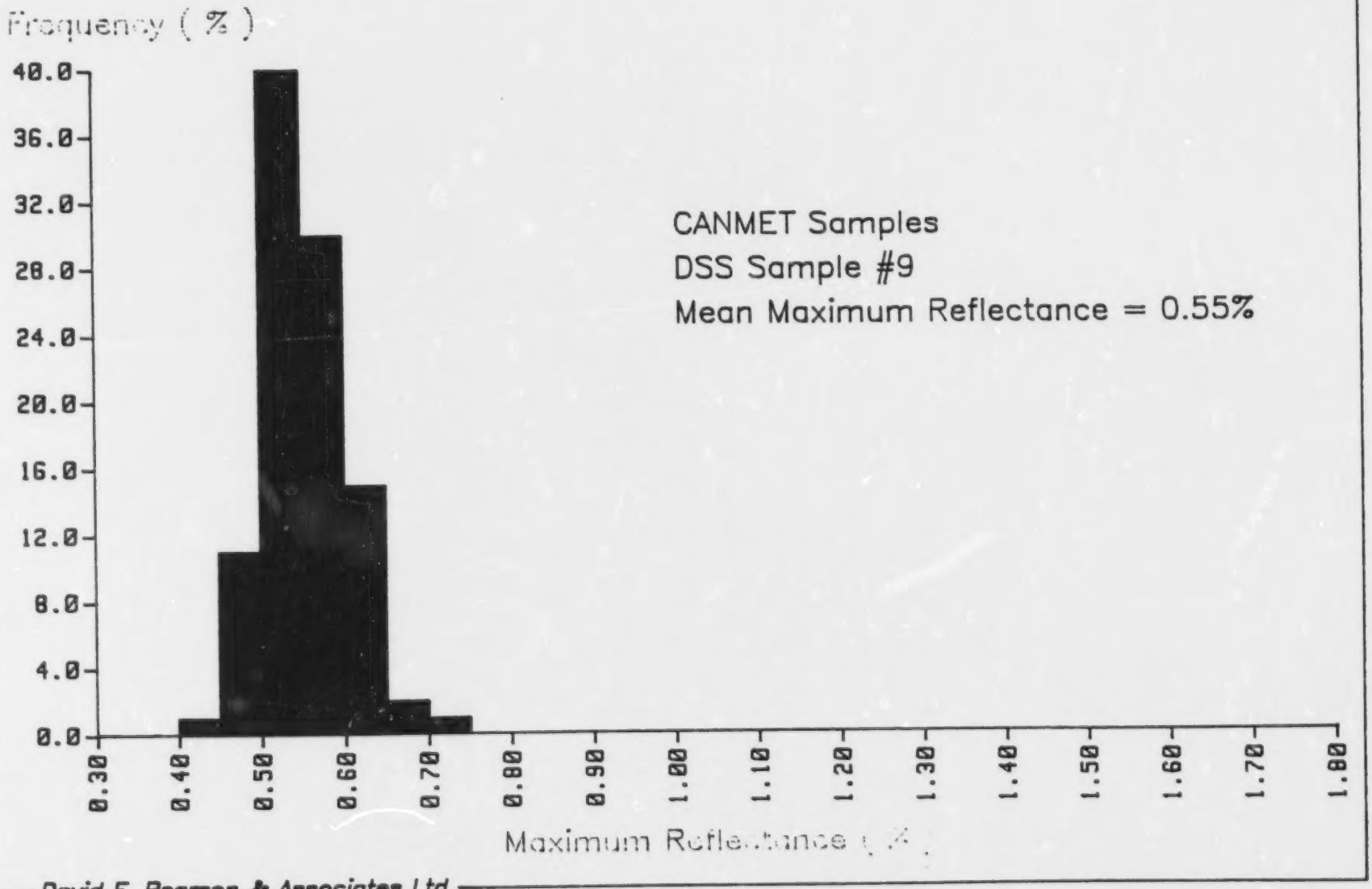
CELL STATISTICS

Cell Number	Lower Limit	Number of Observations	Frequency (%)
3	0.40	1	1.00
4	0.45	11	11.00
5	0.50	40	40.00
6	0.55	30	30.00
7	0.60	15	15.00
8	0.65	2	2.00
9	0.70	1	1.00

VITRINITE TYPE DISTRIBUTION

Vitrinite Type	Frequency (%)
V4	12.00
V5	70.00
V6	17.00
V7	1.00

VITRINITE HISTOGRAM



David E. Pearson & Associates Ltd.

Vitrinite Reflectance

CANMET SAMPLES
DSS Sample #10

BASIC STATISTICS

Total Number of Observations	100
Mean Maximum Reflectance (Romax)...%	.53
Standard Error of the Mean	0.00
Coefficient of Variation...%	6.95
Variance	.0013
Standard Deviation	.0365
Skewness	1.2243
Kurtosis	6.1988

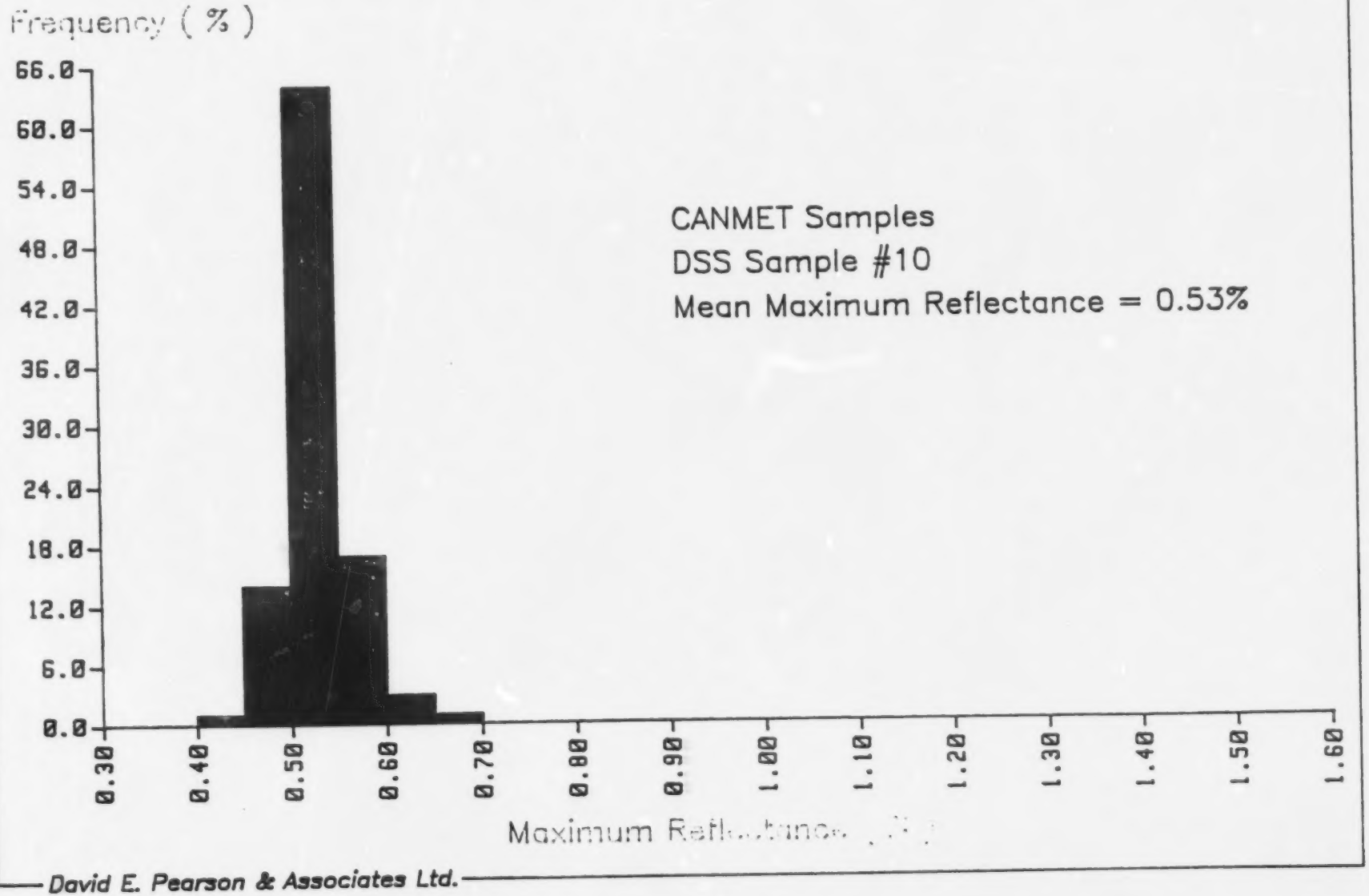
CELL STATISTICS

Cell Number	Lower Limit	Number of Observations	Frequency (%)
3	0.40	1	1.00
4	0.45	14	14.00
5	0.50	64	64.00
6	0.55	17	17.00
7	0.60	3	3.00
8	0.65	1	1.00

VITRINITE TYPE DISTRIBUTION

Vitrinite Type	Frequency (%)
V4	15.00
V5	81.00
V6	4.00

VITRINITE HISTOGRAM



David E. Pearson & Associates Ltd.

Vitrinite Reflectance

CANMET SAMPLES
DSS Sample #11

BASIC STATISTICS

Total Number of Observations	100
Mean Maximum Reflectance (Romax)...%	.63
Standard Error of the Mean	0.00
Coefficient of Variation...%	6.28
Variance	.0016
Standard Deviation	.0397
Skewness	.1645
Kurtosis	3.4597

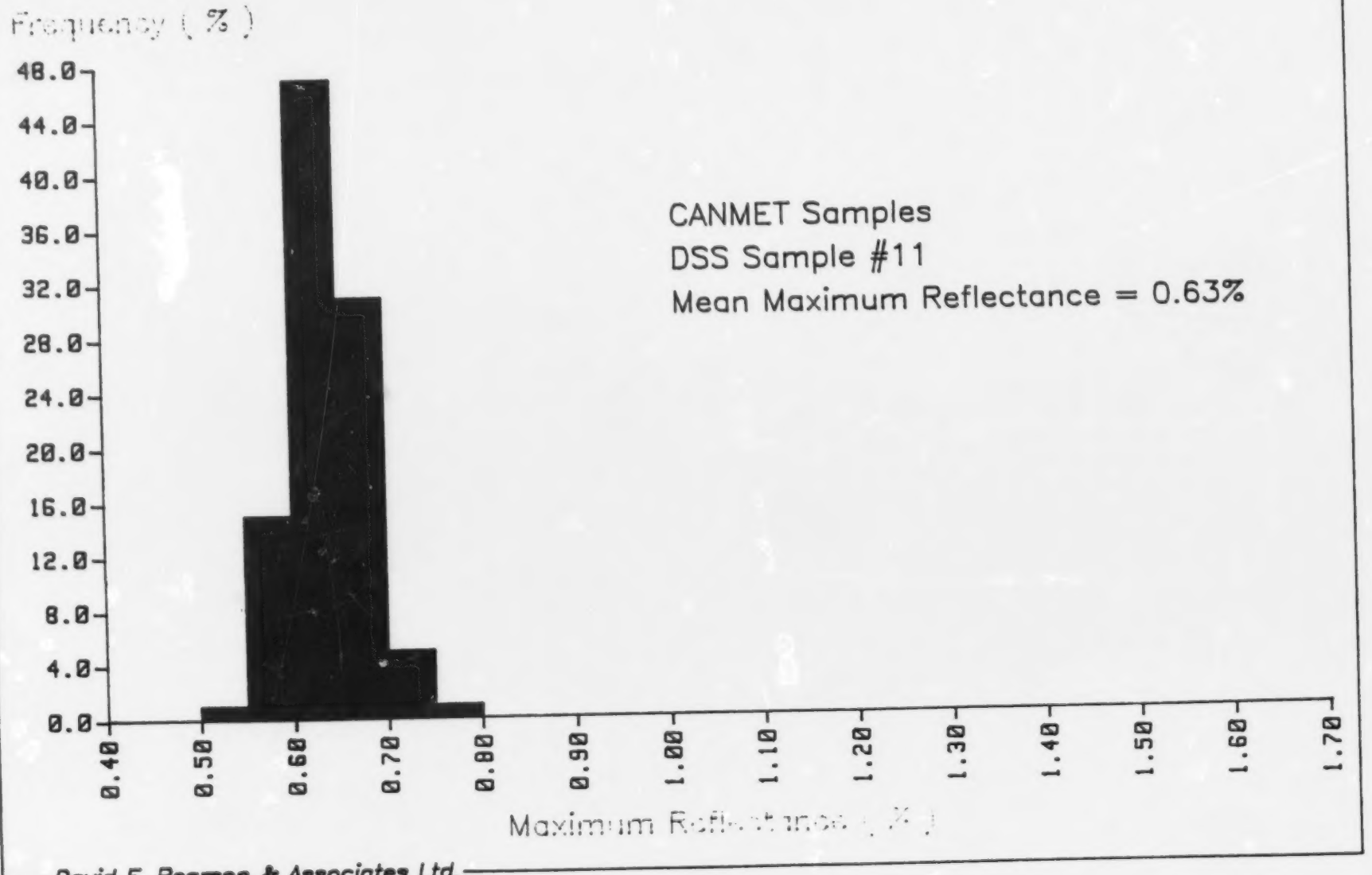
CELL STATISTICS

Cell Number	Lower Limit	Number of Observations	Frequency (%)
3	0.50	1	1.00
4	0.55	15	15.00
5	0.60	47	47.00
6	0.65	31	31.00
7	0.70	5	5.00
8	0.75	1	1.00

VITRINITE TYPE DISTRIBUTION

Vitrinite Type	Frequency (%)
V5	16.00
V6	78.00
V7	6.00

VITRINITE HISTOGRAM



David E. Pearson & Associates Ltd.

Vitrinite Reflectance

CANMET SAMPLES
DSS Sample #12

BASIC STATISTICS

Total Number of Observations	100
Mean Maximum Reflectance (Romax)...%	.92
Standard Error of the Mean	.01
Coefficient of Variation...%	13.65
Variance	.0157
Standard Deviation	.1254
Skewness	-1.8525
Kurtosis	5.6311

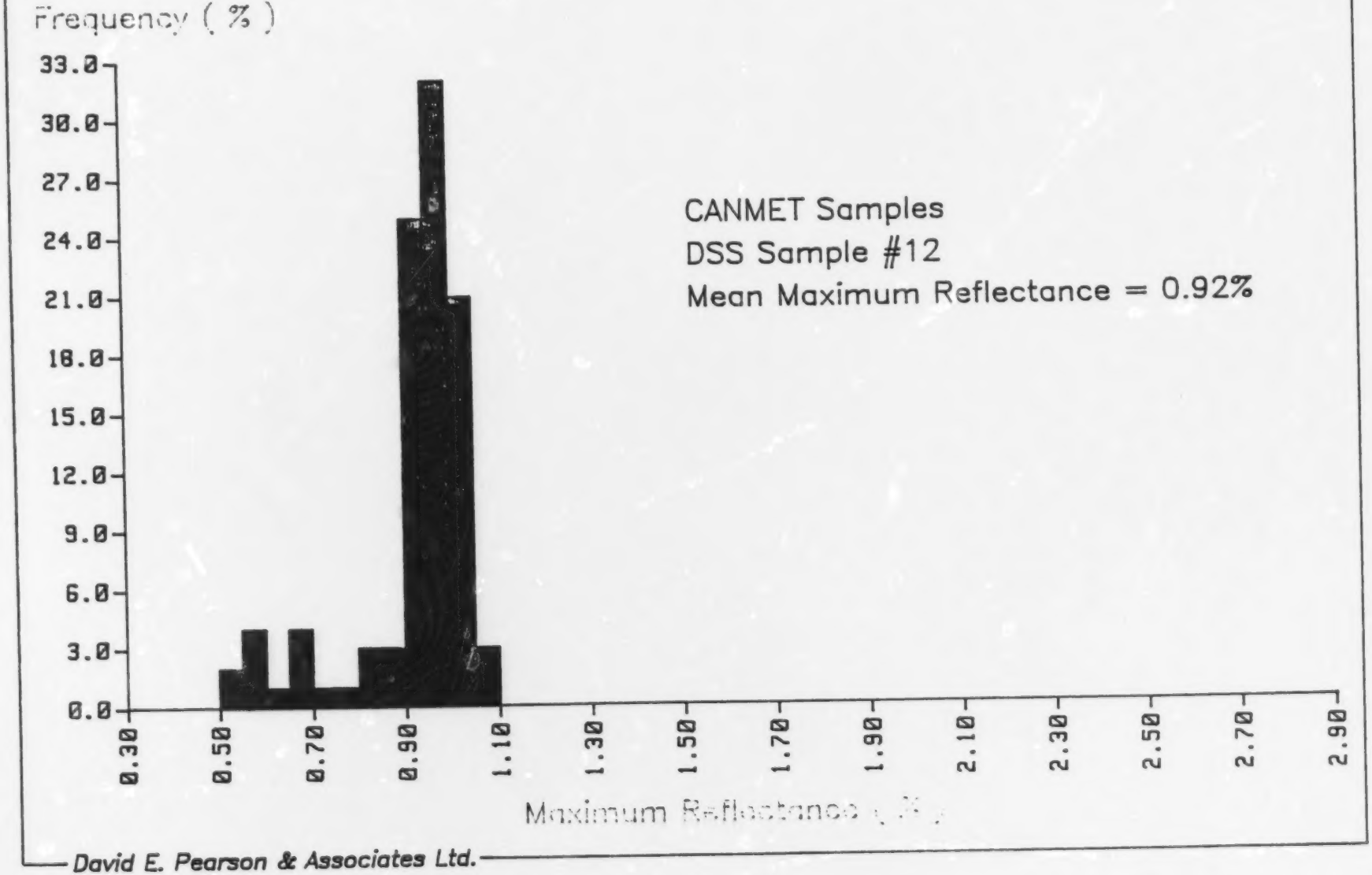
CELL STATISTICS

Cell Number	Lower Limit	Number of Observations	Frequency (%)
5	0.50	2	2.00
6	0.55	4	4.00
7	0.60	1	1.00
8	0.65	4	4.00
9	0.70	1	1.00
10	0.75	1	1.00
11	0.80	3	3.00
12	0.85	3	3.00
13	0.90	25	25.00
14	0.95	32	32.00
15	1.00	21	21.00
16	1.05	3	3.00

VITRINITE TYPE DISTRIBUTION

Vitrinite Type	Frequency (%)
V5	6.00
V6	5.00
V7	2.00
V8	6.00
V9	57.00
V10	24.00

VITRINITE HISTOGRAM



David E. Pearson & Associates Ltd.

CYCLONE ENGINEERING
SALES LTD.

MICROMEDIA

CONTRACT SERIAL NUMBER 69139-01-SQ

DSS REQUISITION NUMBER 23440-6-9139

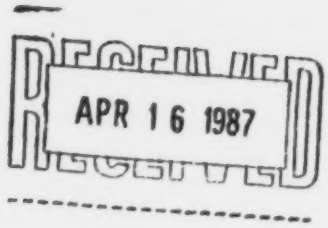
CONTRACTOR: CYCLONE ENGINEERING SALES

FINAL REPORT
Prepared for
DSS 23440-6-9139101ISQ
by
Alberta Research Council
(Porosity, Surface Area and Pore
Size Distribution)

CYCLONE ENGINEERING SALES LTD.
9719 - 60 Avenue
Edmonton, Alberta
T6E 0C4

Telephone: (403)436-1385
Telex: 037-3793
Fax: (403)436-4103

ALBERTA
RESEARCH
COUNCIL



V/P

April 13, 1987

Mr. V. Patel
Cyclone Engineering Sales Ltd.
9719 - 60 Avenue
Edmonton, Alberta
T6E OC4

Dear Mr. Patel:

Enclosed is the analysis report referring to your order No. 2667, dated February 25, 1987.

An invoice will be forwarded to you under separate cover by our Finance Department.

Yours truly,

Sujit Chakrabartty
Coal Research Department

SC/pdt
Encl.
cc: S. Stedman

Energy Resources Division

Coal Research Department
1 Oil Patch Drive
Devon, Alberta
403/987-8111 Telex 037-2147
Mailing address:
P.O. Bag #1310
Devon, Alberta, Canada T0C 1E0

Interim High Pressure Research Facility
1901 - Fifth Street
Nisku, Alberta
403/955-7891 Telex 037 2147
Mailing address
P.O. Box #429
Nisku, Alberta, Canada T0C 2G0

SURFACE CHARACTERISTICS OF CANADIAN COALS

POROSITY, SURFACE AREA AND PORE SIZE DISTRIBUTION

D.S.S. Contract

CYCLONE ENGINEERING SALES LTD.
9719 - 60 Ave.
Edmonton, Alta. T6E 0C4

Subcontracted to

Alberta Research Council through
Purchase Order No. 2667.
Dated February 25, 1987.

Report Contact: Sujit Chakrabartty
Senior Research Officer

Date of Submission: April 15, 1987

Analytical Laboratory
Coal Department
Alberta Research Council
Coal Research Centre
Devon, Alberta.

TABLE OF CONTENTS

1. Experimental Procedure

2. Sample Preparation (Predrying)

3. Standard Method of Analysis

4. Results:
   (a) Table 1. Average Surface Area and Cumulative Pore Volumes of 12 D.S.S. Samples

   (b) Table 2. Average Surface Area and Cumulative Pore Volumes of Standard Specimens for Comparison

   (c) Figures of Adsorption Isotherms

   (d) Figures of Pore Size Distribution

EXPERIMENTAL PROCEDURE

The Micromeritics Digisorb 2600 was used to measure all Cyclone Engineering samples and the standard specimens.

The Micromeritics Digisorb 2600 measures the amount of gas adsorbed and/or desorbed by a sample to calculate the specific surface area, pore volume and pore area distribution, and pore area vs pore diameter. The instrument is an integrated system with key controls and access points arranged for user convenience.

The operator communicates with the computer through a terminal answering posed questions. An entrance manifold is employed to select the desired gases and deliver them to a sample manifold for either sample preparation or analysis. Gas pressure is measured by pressure transducers. The entrance manifold may be evacuated by the vacuum system when required to prevent gas mixing. The vacuum system also serves to evacuate the sample preparation manifold. Five independently controlled ovens heat the samples as required during preparation.

The vacuum system is capable of evacuating essential components to <10-4 mmHg. This is accomplised by a mechanical forepump and a high performance air cooled diffusion pump interconnected through a large bore, pneumatically operated, bellows valve. This valve is controlled by a selenoid valve. A microswitch on the bellows valve signals valve position to a display panel light and the computer.

Two independent pressure gauges are used in the Digisorb 2600. Capacitance-type pressure sensors are employed for the higher pressure measurements and a Penning vacuum gauge is used for measuring the low pressures generated by the vacuum system.

The Penning vacuum gauge measures pressure as a function of ion flow from a high potential electrode to the surrounding grounded tube within a magnetic field. The Penning gauge is used to measure pressures from 0.025 mmHg to less than 10-4 mmHg.

Pressure measurements at higher levels are made using two, zero reference, capacitance-type pressure sensors. The zero reference is a sealed high vacuum cavity. Pressure on a diaphragm changes the capacitance which is converted to a pressure value by a sensitive electronic measurement system.

SAMPLE PREPARATION: (Predrying)

The samples were dried in a vacuum drying oven for two hours at 70 C and 685 mmHg. They were then degassed in the sample degassing ports of the instrument at 105 C for ten hours after being flushed with helium.

STANDARD METHOD OF ANALYSIS

An automatic analysis involves a seris of operations described below.

The sample has been weighed after degassing on the Digisorb 2600 and the weighing data and any other pertinent data have been entered.

1. The manifold is backfilled with helium and all samples are evacuated to 0.025 mmHg. Evacuation continues for 4 hours.

2. Samples are tested individually for leaks or excessive outgassing exceeding 5 micro-m/min. Sample is aborted if it exceeds this rate.

3. PO tube is evacuated and filled with nitrogen. The dewar flask is raised. Saturation pressure PO will be determined by assuring condensation in the PO tube and determining the equilibration pressure once the level of liquid nitrogen in the flask has stabilized.

4. Three free space measurements are made for each analysis sample through measured doses of helium. The three measurements, the average, and the standard deviation are printed.

5. The manifold is backfilled with helium and all analysis samples are evacuated to 0.0005 mmHg.

6. The sample is then analyzed by dosing in nitrogen to the pressures specified by the relative pressures from the dialogue and the current PO reading. PO is updated every 120 minutes.

7. Pressure and volume adsorbed data will be stored for the report when pressures reach to within 5% or when adsorption since the last point has reached 100 cc STP/g.

8. A final PO measurement is taken and stored when all the adsorption and desorption data points for the sample have been taken. The report is printed at this time.

   The sequence then returns to step 6 for the next analysis sample. When all samples are completed the sequence proceeds as follows.

9. The manifold is backfilled with helium and all samples are evacuated to 0.1 mmHg. The PO tube is evacuated to 0.1 mmHg. The dewar flask is lowered.

10. All samples are backfilled to 850 mmHg with helium.

11. The manifold is evacuated, RELOAD is displayed and the 2600 enters the manual mode.

12. The analysis is complete.

NOTE: The results reported on the following pages are based on adsorption data points only. A few samples were analyzed twice to confirm the repeatability of the experiments.

TABLE 1. SURFACE AREA AND CUMULATIVE PORE VOLUME

INSTRUMENT: Micromeritics Surface Area Analyzer - Model 2600

Sample Identification #	B.E.T Surface Area (m2/g) dry	Cumulative Pore Volume (cc/g) dry
DSS Coal - Cyclone 1	1.9687*	0.005026
DSS Coal - Cyclone 2	20.8764	0.031459
DSS Coal - Cyclone 3	5.3430	0.012011
DSS Coal - Cyclone 4	3.2963	0.010511
DSS Coal - Cyclone 5	1.8389	0.009491
DSS Coal - Cyclone 6	11.5602	0.020851
DSS Coal - Cyclone 7	2.9702	0.010277
DSS Coal - Cyclone 8	2.9861	0.009853
DSS Coal - Cyclone 9	2.5752	0.011015
DSS Coal - Cyclone 10	5.7056	0.015791
DSS Coal - Cyclone 11	3.1564*	0.009080
DSS Coal - Cyclone 12	3.1830	0.012895

* - Langmuir Surface Area values.

TABLE 2. CALIBRATION DATA: SURFACE AREA OF STANDARD MATERIALS

	Reported Data		Determined Data using Model 2600	
Sample Specimen	B.E.T. S.A. (m2/g)	Cum. Pore Vol. (cc/g)	B.E.T. S.A. (m2/g)	Cum. Pore Vol. (cc/g)
Vesta Coal (Alta. Subbit.)	2.85	0.01122	2.918	0.02112
Alumina (Quantachrome)	109	---	110.811	0.28837
Alumina (Duke Scientific)	1.4	---	1.443 1.0497*	0.00478 0.00283*

* - Langmuir Surface Area values.

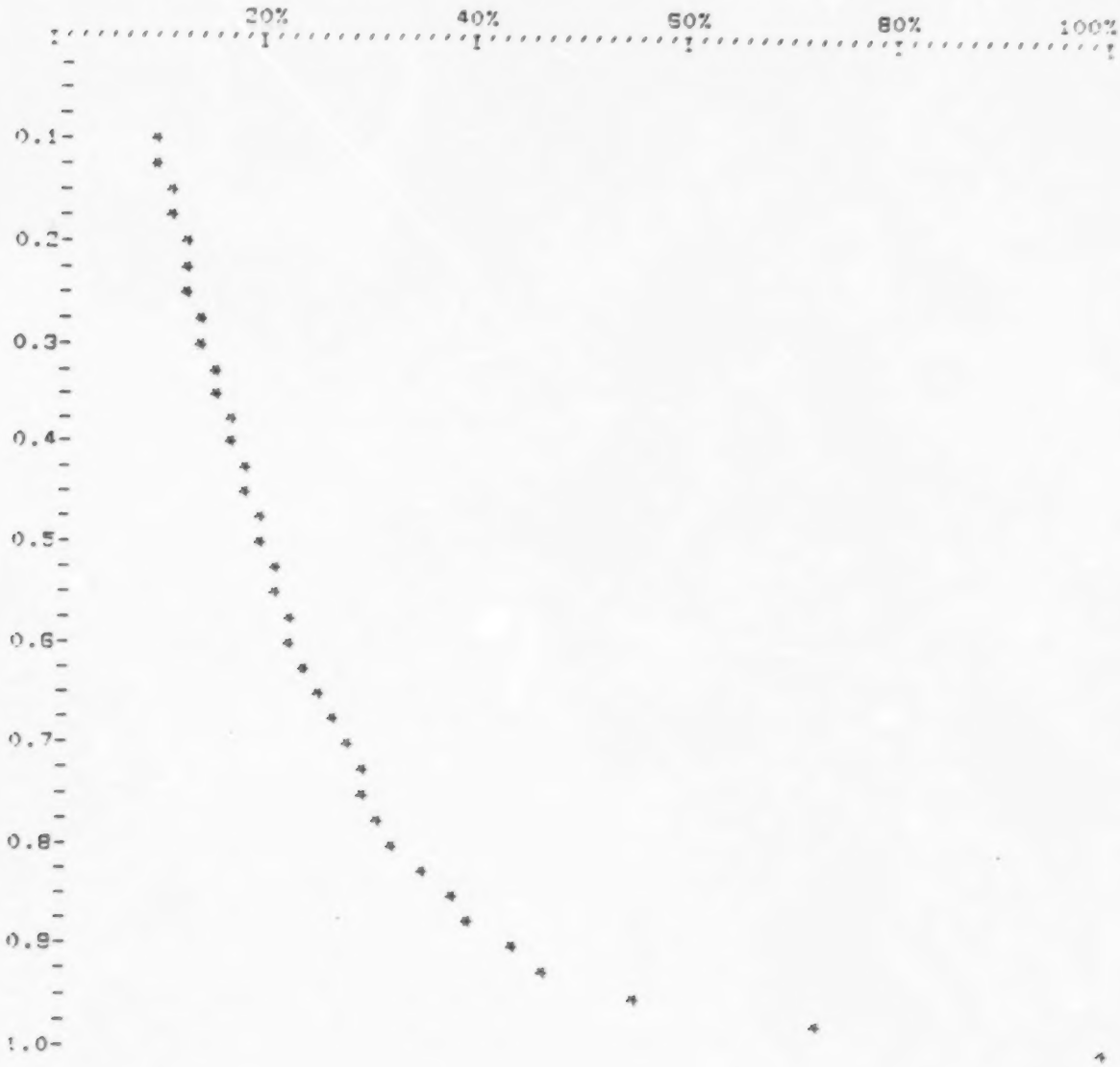
ALBERTA RESEARCH COUNCIL

DIGISORB 2500 ARCHIVE V1.01 PAGE 1
105

ALUMINA STAN. S.A.=1.4
STATION 1 STARTED 4/ 2/87 9:21 COMPLETED 4/ 2/87 16:23 NITROGEN

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE
MAX VOL SORBED = 3.0638 CC/G STP MAX VOL FOR PLOT = 3.0638 CC/G STP



ALUMINA STAN. S.A.=1.4 NITROGEN
STATION 1 STARTED 4/ 2/87 9:21 COMPLETED 4/ 2/87 16:23

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.000581 CC/G MAX INC FOR PLOT = 0.000581 CC/G

```
                20%          40%          60%          80%         100%
     I'''''''''''I''''''''''''I''''''''''''I''''''''''''I''''''''''''I
  14.0 ****
  16.5 *****
  19.4 ***********
  22.8 ************************
  26.8 ********************
  31.6 ******************
  37.1 ****************
  43.7 ******************
  51.4 ********************
  60.5 **************************
  71.2 ********************
  83.8 ****************
  98.6 ****************
 116.0 ***************************
 136.5 ************************
 160.6 ********************
 188.9 ******************
 222.3 ******************
 261.6 ************************
 307.8 ****************************
 362.1 ********************************
 426.1 ********************************
 501.3 ****************************
 589.9 ************************
 694.1 ****************************
 816.6 ****************************************
 960.9 ************************************************************
1130.6 ************************************************************************
1330.3 ************************************************************************
1565.2
1841.7
2167.0
2549.7
3000.0
```

ALBERTA RESEARCH COUNCIL

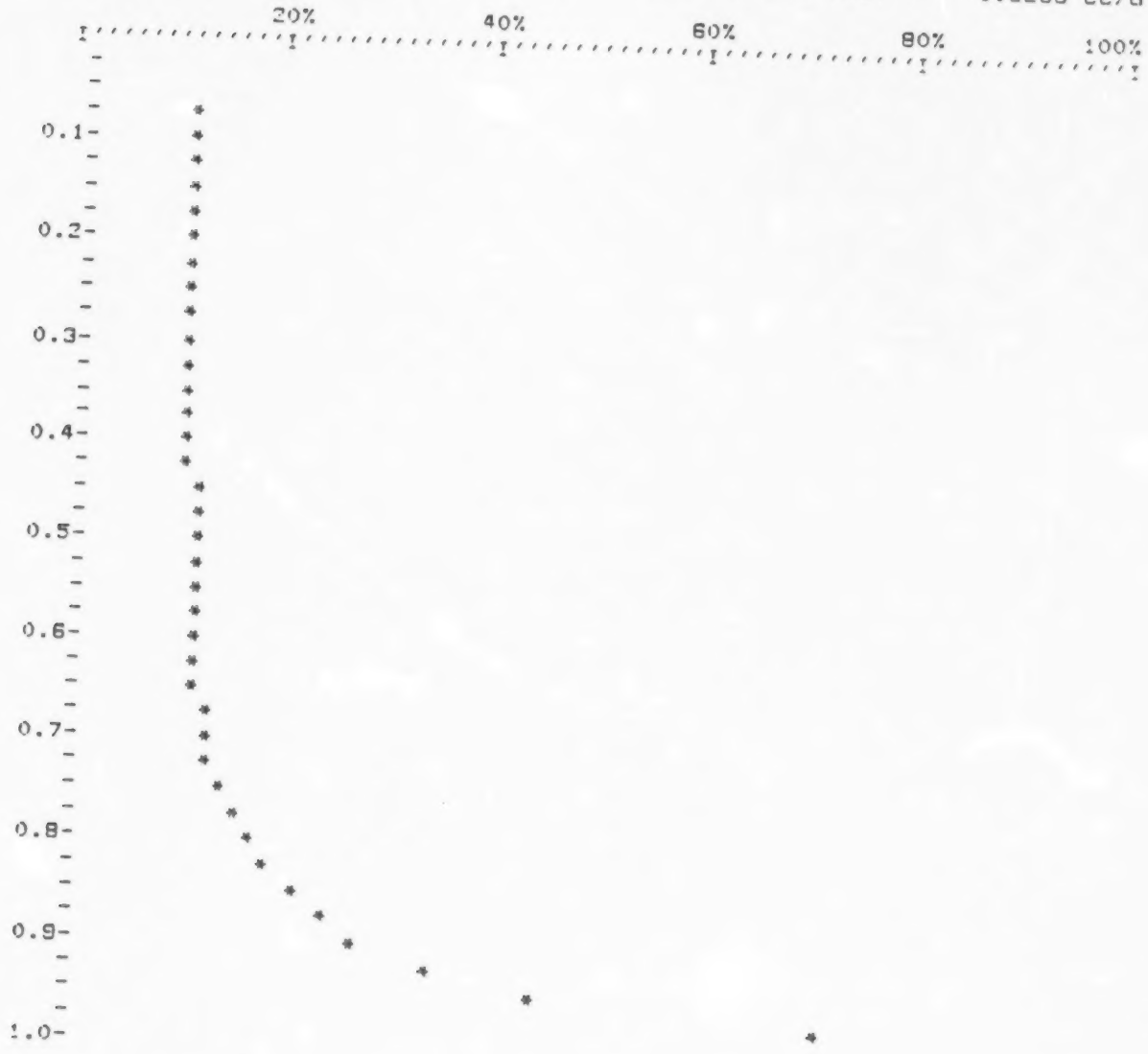
DIGISORB 2600 V3.03
105

ALUMINA S.A.=1.4M2/G
STATION 5 STARTED 4/10/87 8:23 COMPLETED 4/11/87 1:16 NITROGEN

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE

MAX VOL SORBED = 1.9283 CC/G STP MAX VOL FOR PLOT = 1.9283 CC/G STP



ALUMINA S.A.=1.4M2/G NITROGEN
STATION 5 STARTED 4/10/87 8:23 COMPLETED 4/11/87 1:16

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.000485 CC/G MAX INC FOR PLOT = 0.000485 CC/G

```
                20%          40%          60%          80%          100%
    I.............I............I............I............I............I
  14.0
  16.5 **
  19.4
  22.8
  26.8
  31.6
  37.1
  43.7
  51.4
  60.5 ***
  71.2 *******
  83.8 *********
  98.6 ***********
 115.0 ***************
 136.5 ****************
 160.6 ***************
 198.9 ****************
 222.3 ********************
 261.6 ************************
 307.8 ***************************
 362.1 ******************************
 426.1 ***********************************
 501.3 ************************
 589.9 ************************
 694.1 ****************************************
 815.6 ************************************************
 960.9 ********************************************************************
1130.6
1330.3
1565.2
1841.7
2167.0
2549.7
3000.0
```

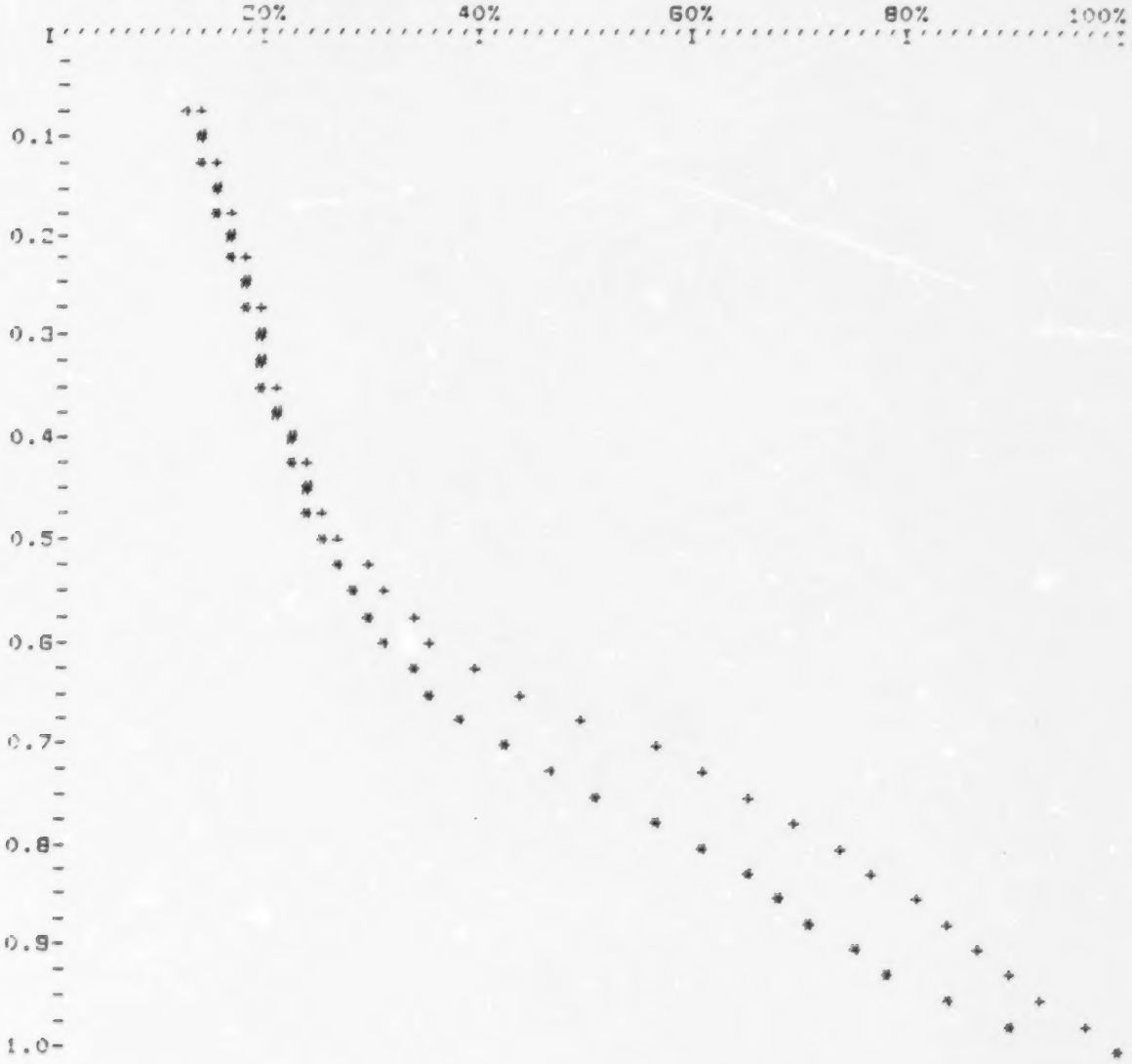
ALBERTA RESEARCH COUNCIL

DIGISORB 2600 ARCHIVE V1.01 PAGE 1
105

ALUMINA STAN. S.A.=109 NITROGEN
STATION 1 STARTED 3/30/87 9:46 COMPLETED 3/31/87 18:16

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE
MAX VOL SORBED = 183.2690 CC/G STP MAX VOL FOR PLOT = 183.2690 CC/G STP



ALUMINA STAN. S.A.=109 NITROGEN
STATION 1 STARTED 3/30/87 9:46 COMPLETED 3/31/87 18:16

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.042373 CC/G MAX INC FOR PLOT = 0.042373 CC/G

```
                20%          40%          60%          80%          100%
    I'''''''''''I''''''''''''I''''''''''''I''''''''''''I''''''''''''I
  14.0
  16.5
  19.4 **
  22.8 *********
  26.8 ***************
  31.6 ********************
  37.1 ********************
  43.7 ************************
  51.4 ****************************************
  60.5 ********************************************************************
  71.2 ************************************************************************
  83.8 ********************************************************************
  98.6 ************************************************
 116.0 ****************************************
 136.5 ***************************
 160.6 ***************
 188.9 ****************
 222.3 ******************
 261.6 ***************
 307.8 ************
 362.1 ************
 426.1 *******
 501.3 ******
 589.9 *******
 694.1 *********
 816.6 ********
 960.9 *********
1130.6 ***************************
1330.3
1565.2
1841.7
2167.0
2549.7
3000.0
```

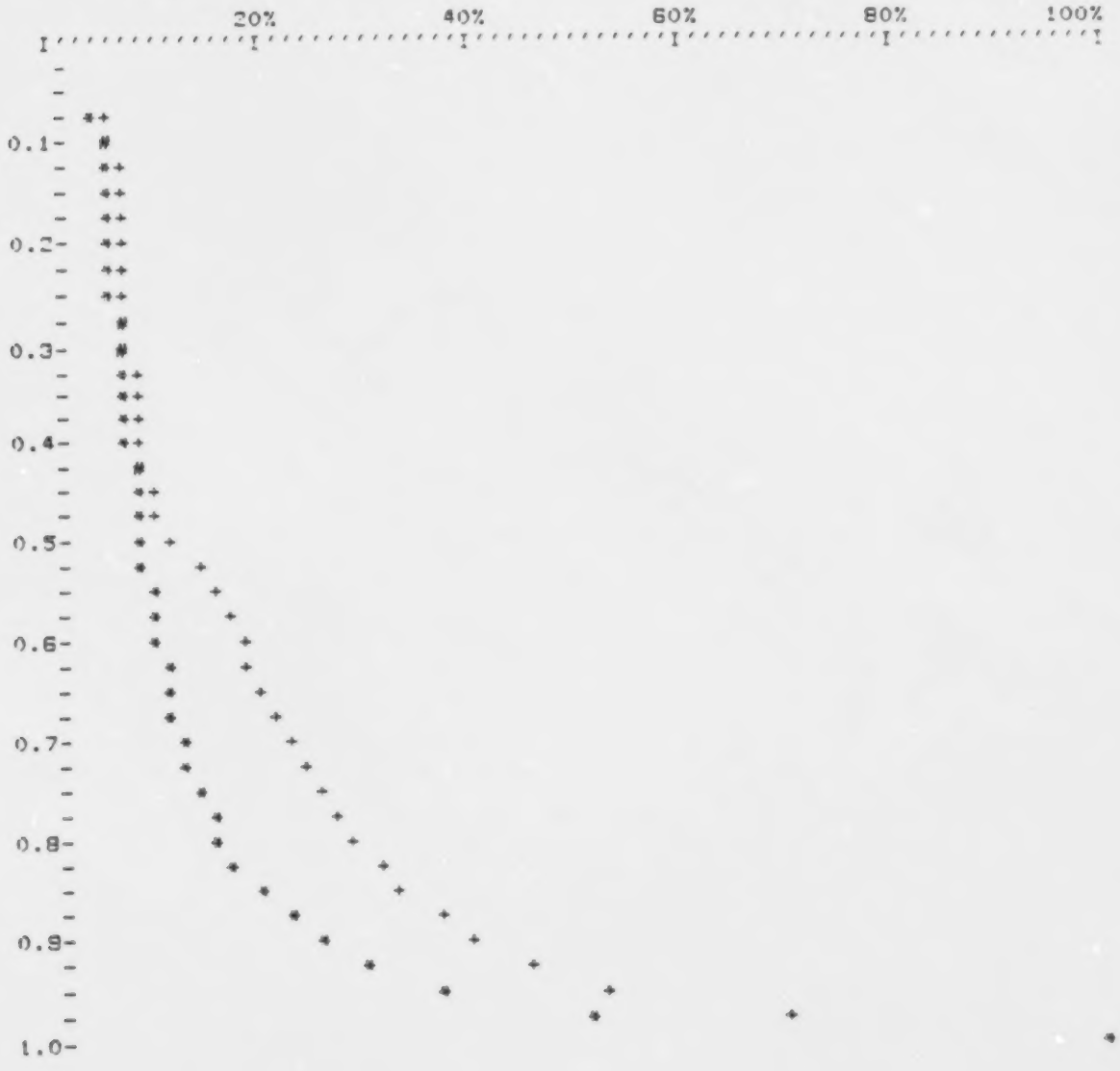
ALBERTA RESEARCH COUNCIL

DIGISORB 2500 ARCHIVE V1.01 PAGE 1
:05

39-97 VESTA ORG. COAL NITROGEN
STATION 4 STARTED 3/30/97 9:46 COMPLETED 4/ 1/87 13: 3

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE
MAX VOL SORBED = 13.7395 CC/G STP MAX VOL FOR PLOT = 13.7395 CC/G STP



39-87 VESTA ORG. COAL NITROGEN
STATION 4 STARTED 3/30/87 9:46 COMPLETED 4/ 1/87 13: 3

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.008258 CC/G MAX INC FOR PLOT = 0.008258 CC/G

```
                20%          40%          60%          80%         100%
    I'''''''''''I''''''''''''I''''''''''''I''''''''''''I''''''''''''I
 14.0
 16.5
 19.4
 22.8 *
 26.8 *
 31.6 **
 37.1 **
 43.7 **
 51.4 ***
 60.5 ***
 71.2 ***
 83.8 ****
 98.6 ****
116.0 *****
136.5 *****
160.6 ******
188.9 ******
222.3 ******
261.6 ********
307.8 *******
362.1 ******
426.1 *******
501.3 ****
589.9 ******
694.1 ******
816.6 ************
960.9 *************************
1130.6 ************************************************
1330.3
1565.2
1841.7
2167.0
2549.7
3000.0
```

ALBERTA RESEARCH COUNCIL

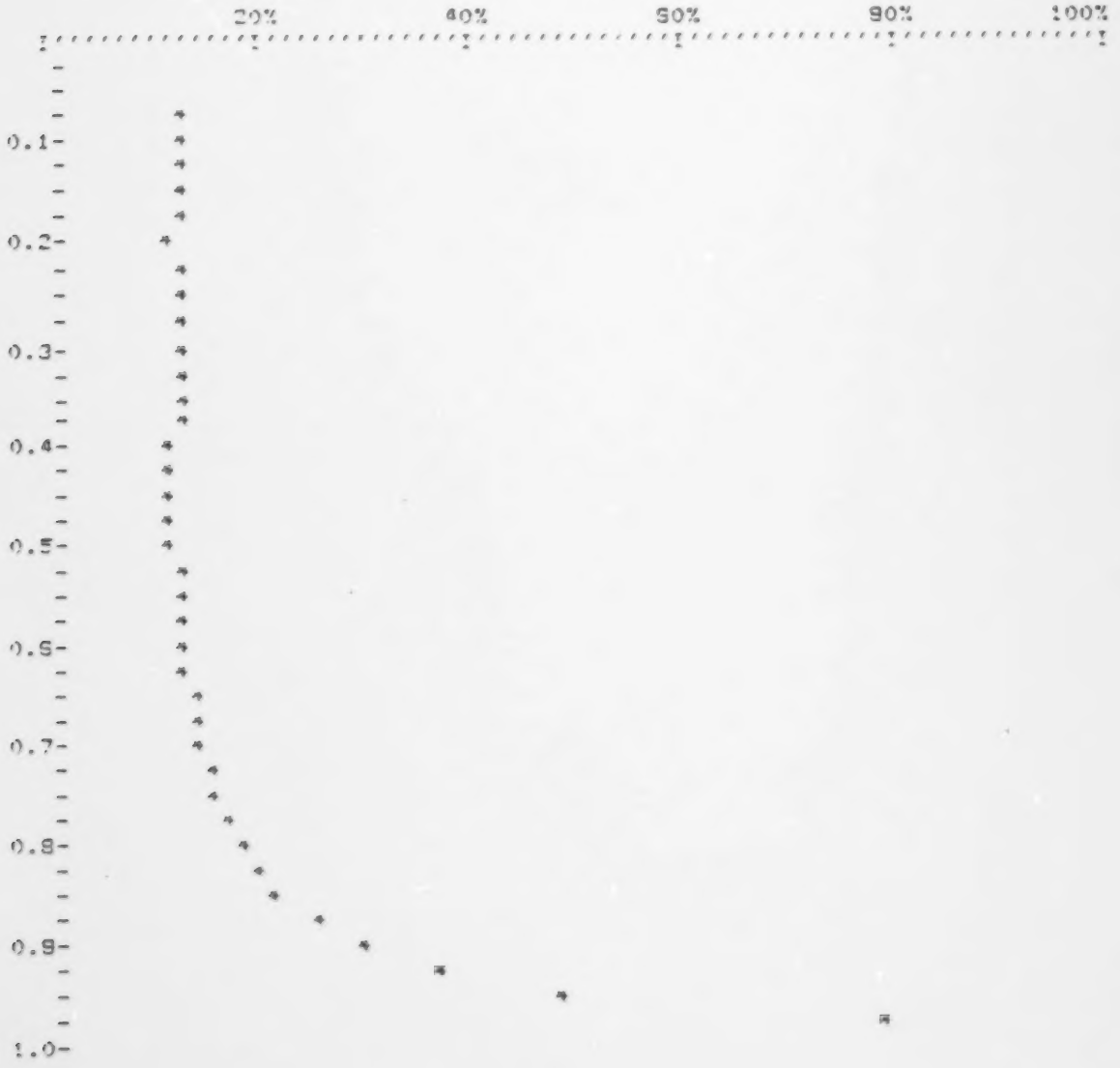
DIGISORB 2500 V3.03
105

15-97 DSS CYCLONE 1 NITROGEN
STATION 1 STARTED 4/10/87 8:23 COMPLETED 4/10/87 15:32

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE

MAX VOL SORBED = 3.4640 CC/G STP MAX VOL FOR PLOT = 3.4640 CC/G STP



ALBERTA RESEARCH COUNCIL

DIGISORB 2600 V3.03 PAGE 105

15-87 DSS CYCLONE 1 NITROGEN
STATION 1 STARTED 4/10/87 8:23 COMPLETED 4/10/87 15:32

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.000716 CC/G MAX INC FOR PLOT = 0.000716 CC/G

```
                20%          40%          60%          80%          100%
     I.....I.....I.....I.....I.....I.....I.....I.....I.....I.....I
  14.0
  16.5
  19.4
  22.8
  26.8
  31.6
  37.1
  43.7
  51.4
  60.5
  71.2
  83.8 **************
  98.6 ****************
 116.0 *********
 136.5 ********************
 160.6 ****************************
 188.9 ****************************
 222.3 ****************************
 261.6 ******************************
 307.8 ************************************
 362.1 ******************************************
 426.1 **************************************************
 501.3 ************************************
 589.9 ************************************
 694.1 ******************************************
 816.6 ************************************************************************
 960.9 ********************************************************************************
1130.6
1330.3
1565.2
1841.7
2167.0
2549.7
3000.0
```

ALBERTA RESEARCH COUNCIL

DIGISORB 2600 ARCHIVE V1.01 PAGE 1
105

16-87 DSS CYCLONE 2 NITROGEN
STATION 2 STARTED 4/ 2/87 9:21 COMPLETED 4/ 2/87 19:50

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE
MAX VOL SORBED = 19.9081 CC/G STP MAX VOL FOR PLOT = 19.9081 CC/G STP

16-87 DSS CYCLONE 2 NITROGEN
STATION 2 STARTED 4/ 2/87 9:21 COMPLETED 4/ 2/87 19:50

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.003397 CC/G MAX INC FOR PLOT = 0.003397 CC/G

```
                20%          40%          60%          80%         100%
    I'''''''''''I''''''''''''I''''''''''''I''''''''''''I''''''''''''I
  14.0 ************
  16.5 ***************
  19.4 ***********************
  22.8 ********************************************************
  26.8 ************************************************************
  31.6 ***************************************************************
  37.1 ********************************************************************
  43.7 ***************************************************************************
  51.4 ********************************************************************
  60.5 ***************************************************************
  71.2 ********************************************************
  83.8 ********************************************
  98.6 ****************************
 116.0 ************************
 136.5 ********************
 160.6 ******************
 188.9 ****************
 222.3 ***************
 261.6 ******************
 307.8 ****************
 362.1 *************
 426.1 ******************
 501.3 *************
 589.9 *********
 694.1 **************
 816.6 ****************
 960.9 ********************
1130.6 ****************************
1330.3
1565.2
1841.7
2167.0
2549.7
3000.0
```

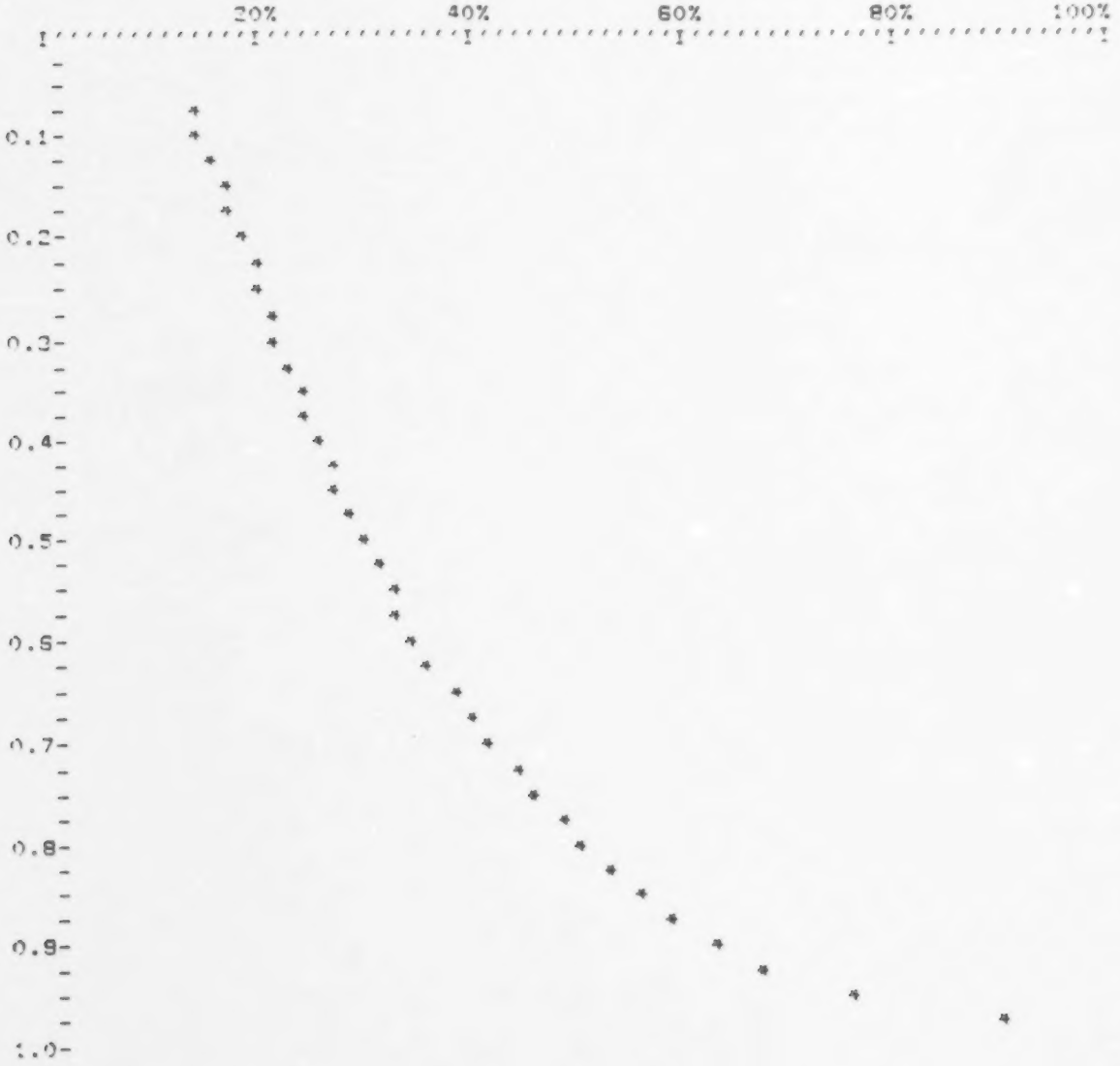
ALBERTA RESEARCH COUNCIL

DIGISORB 2600 ARCHIVE V1.01 PAGE 1
105

17-97 DSS CYCLONE 3 NITROGEN
STATION 3 STARTED 4/ 2/87 9:21 COMPLETED 4/ 2/87 22:12

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE
MAX VOL SORBED = 7.6438 CC/G STP MAX VOL FOR PLOT = 7.6438 CC/G STP



17-97 DSS CYCLONE 3 NITROGEN
STATION 3 STARTED 4/ 2/87 9:21 COMPLETED 4/ 2/87 22:12

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.000794 CC/G MAX INC FOR PLOT = 0.000794 CC/G

```
                20%          40%          60%          80%         100%
     I.....I.....I.....I.....I.....I.....I.....I.....I.....I.....I
  14.0 **************
  16.5 **************
  19.4 ******************************
  22.8 ************************************************************
  26.8 ************************************************************
  31.6 ************************************************************
  37.1 ************************************************************
  43.7 ************************************************************
  51.4 ************************************************************
  60.5 ************************************************************
  71.2 ************************************************************
  83.8 ************************************************************
  98.6 ************************************************************
 116.0 ************************************************************
 136.5 ********************************************************
 160.6 ********************************************************
 188.9 ********************************************************
 222.3 ********************************************************
 261.6 ********************************************************
 307.8 ********************************************************
 362.1 ********************************************************
 426.1 ************************************************************
 501.3 ********************************************
 589.9 ******************************
 694.1 ********************************************
 816.6 ****************************************
 960.9 ********************************************
1130.6
1330.3
1565.2
1841.7
2167.0
2549.7
3000.0
```

ALBERTA RESEARCH COUNCIL

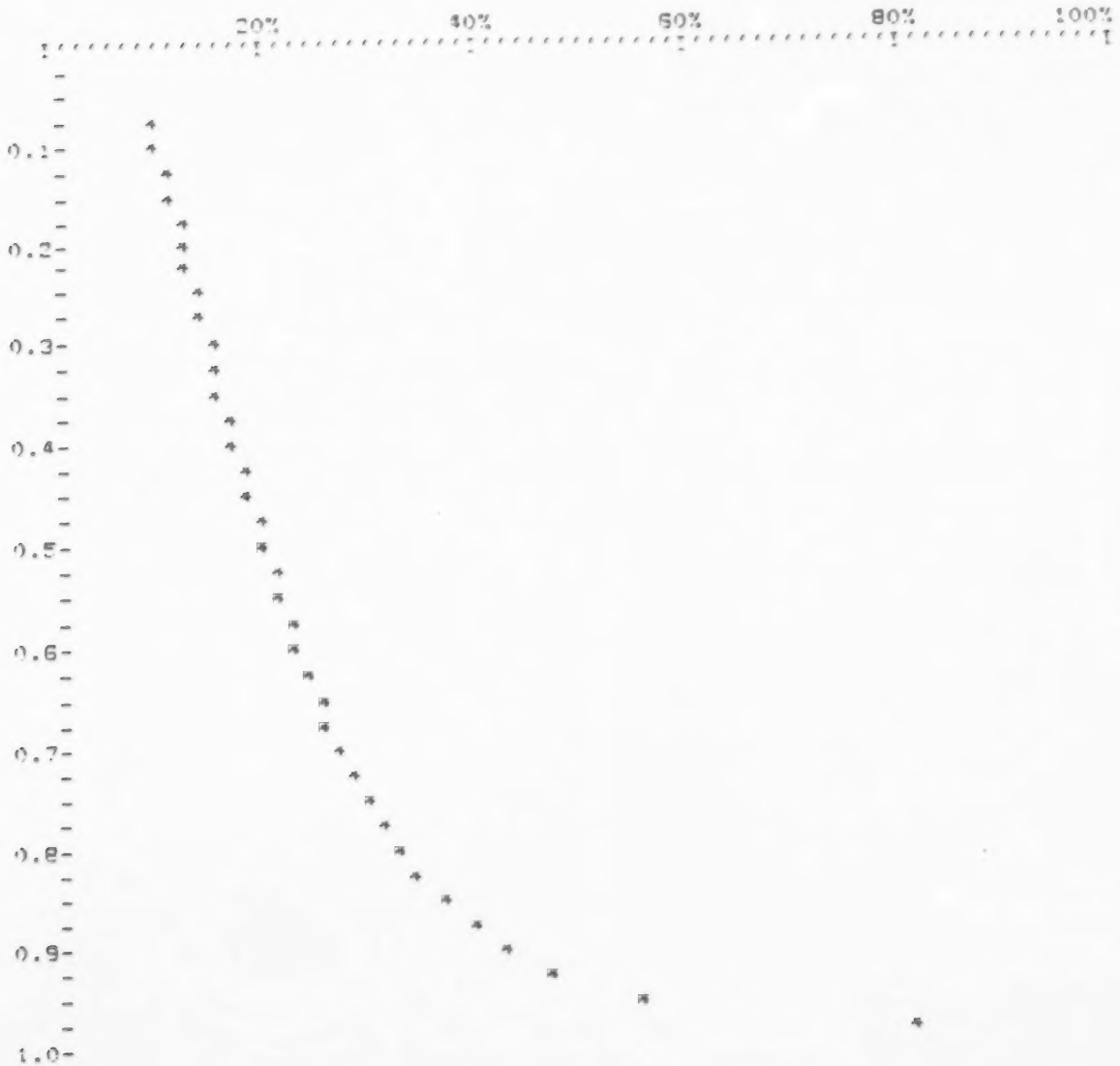
DIGISORB 2600 ARCHIVE V1.01 PAGE 1
105

19-97 DSS CYCLONE 4 NITROGEN
STATION 4 STARTED 4/ 2/87 9:21 COMPLETED 4/ 3/87 0:27

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE

MAX VOL SORBED = 6.8031 CC/G STP MAX VOL FOR PLOT = 6.8031 CC/G STP



18-87 DSS CYCLONE 4 NITROGEN
STATION 4 STARTED 4/ 2/87 9:21 COMPLETED 4/ 3/87 0:27

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.001835 CC/G MAX INC FOR PLOT = 0.001835 CC/G

```
                20%          40%          60%          80%         100%
     I'''''''''''I''''''''''''I''''''''''''I''''''''''''I''''''''''''I
  14.0 ****
  16.5 ****
  19.4 ********
  22.8 ****************
  26.8 **************
  31.6 ************
  37.1 ************
  43.7 ************
  51.4 **************
  60.5 ***************
  71.2 ***************
  83.8 **************
  98.6 ***************
 116.0 ****************
 136.5 ***************
 160.6 ************
 188.9 ************
 222.3 ***************
 261.6 ******************
 307.8 ********************
 362.1 ********************
 426.1 ************************************************
 501.3 ************************************************************
 589.9 ****************
 694.1 ************************************
 816.6 ************************************
 960.9 ************************************
1130.6
1330.3
1565.2
1841.7
2167.0
2549.7
3000.0
```

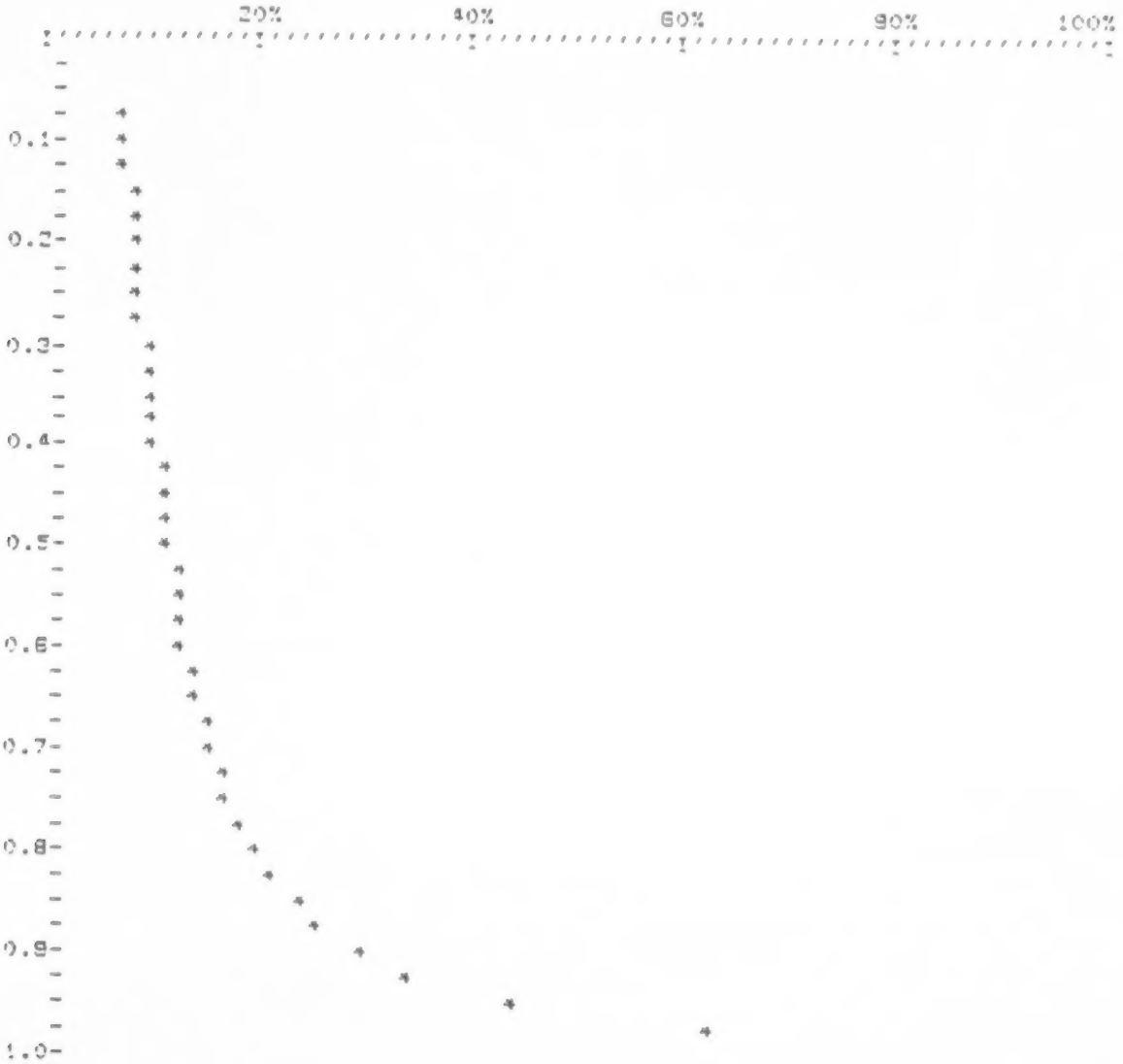
ALBERTA RESEARCH COUNCIL

DIGISORB 2500 V2.03 PAGE 1
105

19-87 DSS CYCLONE 5 NITROGEN
STATION 5 STARTED 4/ 7/87 8:30 COMPLETED 4/ 8/87 2: 4

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE
MAX VOL SORBED = 5.0796 CC/G STP MAX VOL FOR PLOT = 5.0796 CC/G STP



19-87 DSS CYCLONE 5 NITROGEN
STATION 5 STARTED 4/ 7/87 8:30 COMPLETED 4/ 8/87 2: 4

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.002941 CC/G MAX INC FOR PLOT = 0.002941 CC/G

```
                20%          40%          60%          80%         100%
     I'''''''''''I''''''''''''I''''''''''''I''''''''''''I''''''''''''I
  14.0
  16.5 *
  19.4 *
  22.8 **
  26.8 **
  31.6 **
  37.1 ***
  43.7 ***
  51.4 ****
  60.5 ****
  71.2 *****
  83.9 *****
  98.6 ******
 116.0 *******
 136.5 *******
 160.6 *******
 188.9 ********
 222.3 *********
 261.6 **********
 307.8 ************
 362.1 ***************
 426.1 **************
 501.3 **********
 589.9 *******
 694.1 ***********
 816.6 ****************
 960.9 ***********************************
1130.6 ********************************************************************************
1330.3
1565.2
1841.7
2167.0
2549.7
3000.0
```

ALBERTA RESEARCH COUNCIL

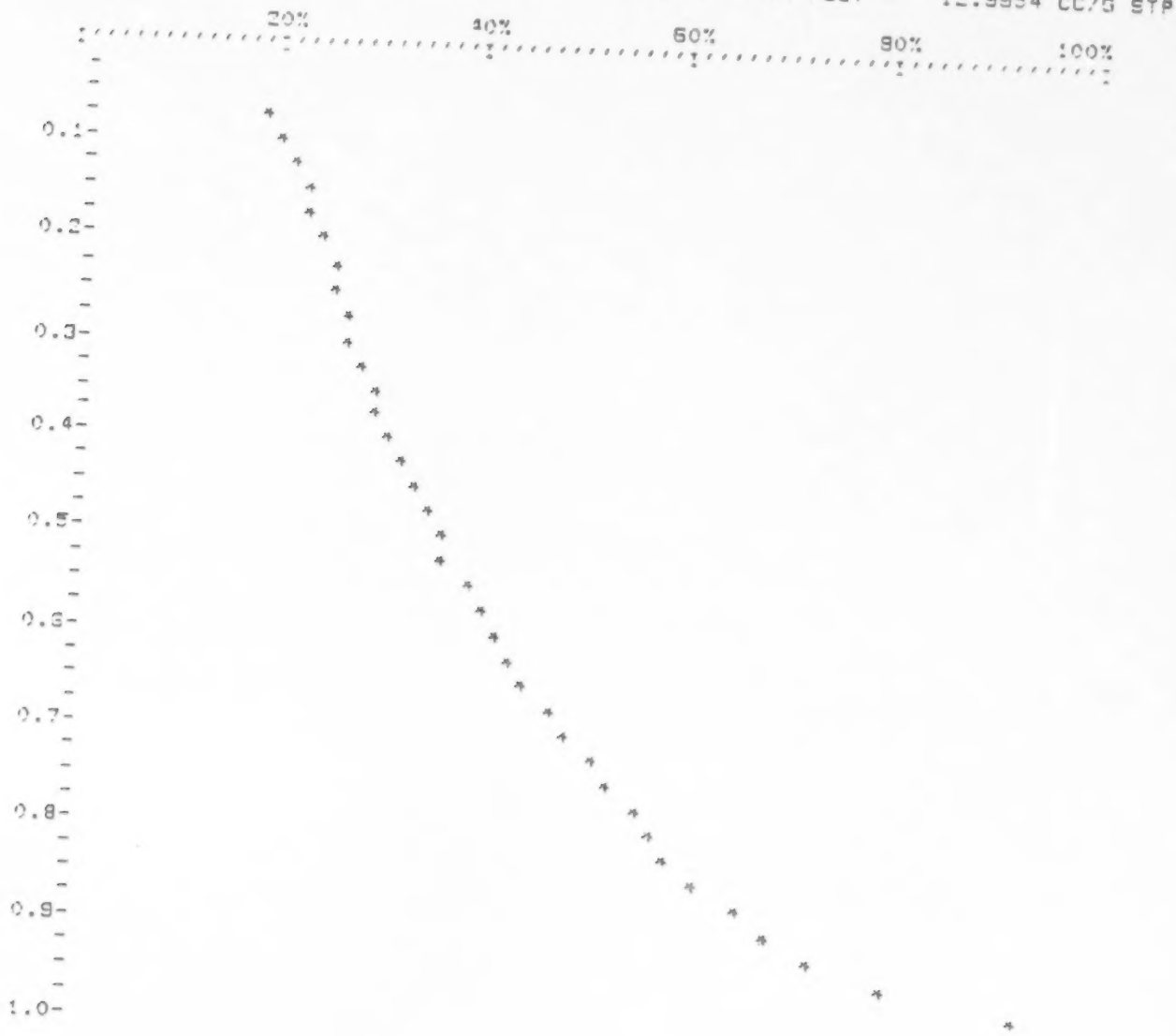
DIGISORB 2500 V3.03
105

20-87 DSS CYCLONE 6
STATION 1 STARTED 4/ 7/87 8:30 COMPLETED 4/ 7/87 15:46 NITROGEN

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE

MAX VOL SORBED = 12.9954 CC/G STP MAX VOL FOR PLOT = 12.9954 CC/G STP



20-87 DSS CYCLONE 6 NITROGEN
STATION 1 STARTED 4/ 7/87 8:30 COMPLETED 4/ 7/87 16:48

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.001728 CC/G MAX INC FOR PLOT = 0.001728 CC/G

```
                20%          40%          60%          80%         100%
              I............I............I............I............I
  14.0
  16.5 ***************
  19.4 ************
  22.8 ************************************************
  26.8 ********************************************************
  31.6 ********************************************************
  37.1 ************************************************************
  43.7 ********************************************************************
  51.4 ************************************************************************
  60.5 ************************************************************************
  71.2 ******************************************************************
  83.8 ********************************************************
  98.6 ****************************************
 116.0 ****************************************
 136.5 ****************************************
 160.6 ****************************************
 188.9 ******************************
 222.3 ******************************
 261.6 ******************************
 307.8 ******************************
 362.1 ********************************
 426.1 ****************************************
 501.3 ******************
 589.9 ***********************
 694.1 ********************************
 816.6 ************************************
 960.9
1130.6
1330.3
1565.2
1841.7
2167.0
2549.7
3000.0
```

ALBERTA RESEARCH COUNCIL

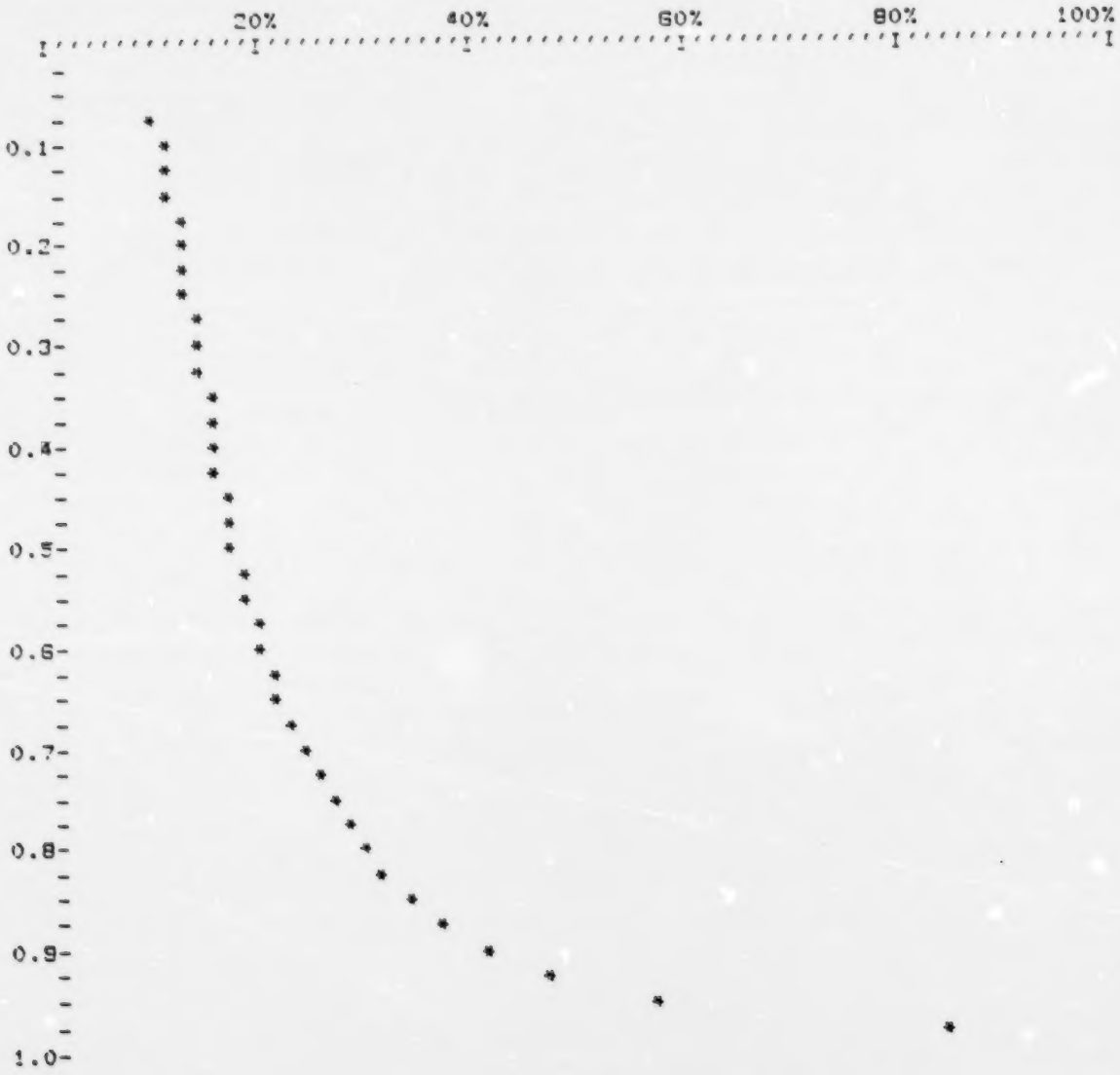
DIGISORB 2600 V3.03 PAGE 1
105

21-87 DSS CYCLONE 7 NITROGEN
STATION 2 STARTED 4/ 7/87 8:30 COMPLETED 4/ 7/87 19: 4

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE

MAX VOL SORBED = 6.5267 CC/G STP MAX VOL FOR PLOT = 6.5267 CC/G STP



ALBERTA RESEARCH COUNCIL

DIGISORB 2600 V3.03 PAGE
105

21-87 DSS CYCLONE 7 NITROGEN
STATION 2 STARTED 4/ 7/87 8:30 COMPLETED 4/ 7/87 19: 4

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.001127 CC/G MAX INC FOR PLOT = 0.001127 CC/

```
                20%          40%          60%          80%         100%
     I'''''''''''I''''''''''''I''''''''''''I''''''''''''I''''''''''''I
  14.0
  16.5 ******
  19.4 ***
  22.8 **********
  26.8 ***************
  31.6 *************
  37.1 ************
  43.7 ***************
  51.4 ********************
  60.5 ************************
  71.2 ************************
  83.9 ********************
  98.6 ********************
 116.0 *************************
 136.5 *************************
 160.6 *************************
 188.9 ******************************
 222.3 ***********************************
 261.6 ***************************************
 307.8 ******************************************
 362.1 ***********************************************
 426.1 ***************************************************
 501.3 ***********************************
 589.9 ******************************
 694.1 ************************************************
 816.6 ********************************************************
 960.9 ****************************************************************
1130.6
1330.3
1565.2
1841.7
2167.0
2549.7
3000.0
```

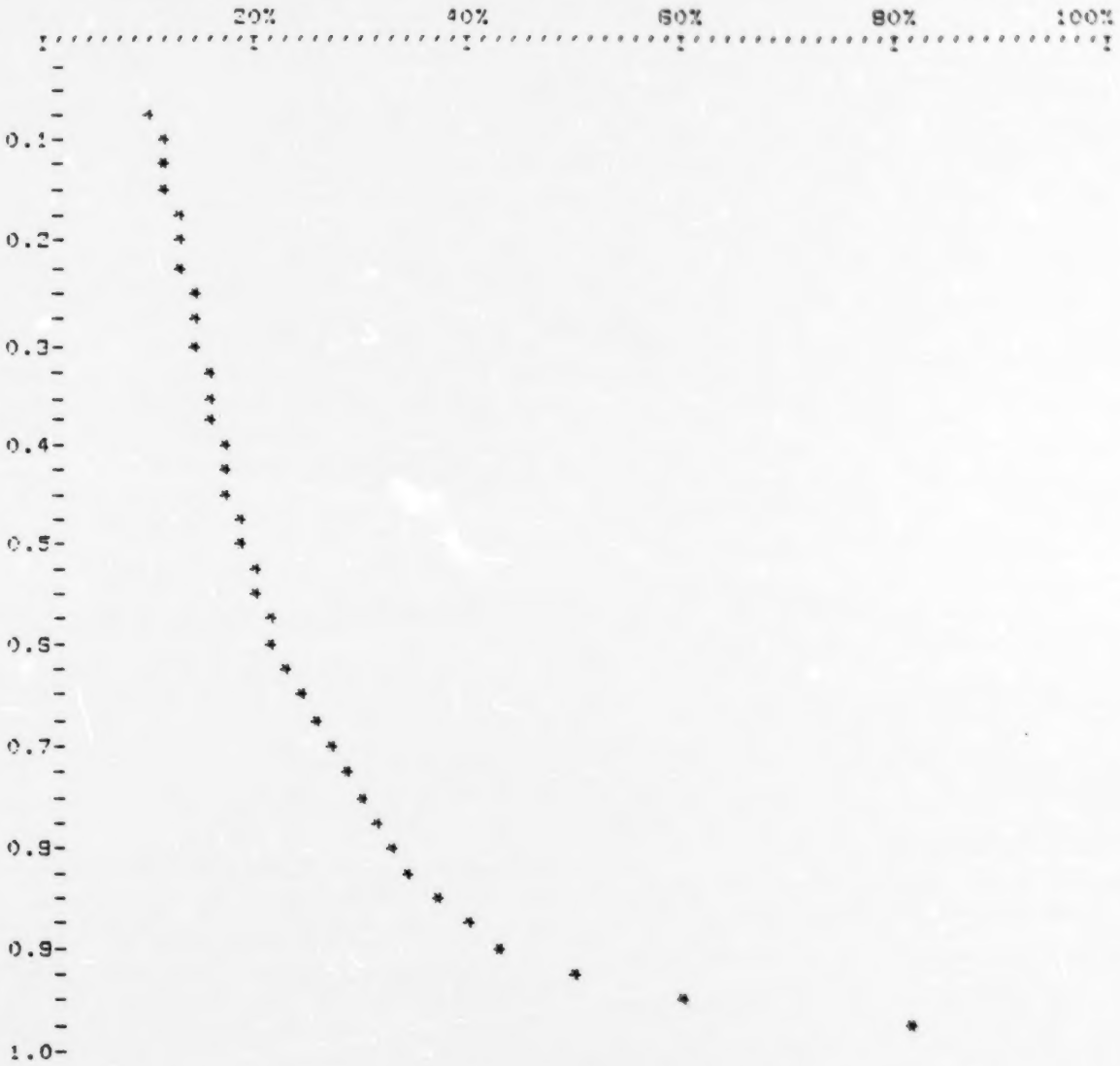
ALBERTA RESEARCH COUNCIL

DIGISORB 2600 V3.03 PAGE 1
105

22-97 DSS CYCLONE 8 NITROGEN
STATION 3 STARTED 4/ 7/87 8:30 COMPLETED 4/ 7/87 21:22

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE
MAX VOL SORBED = 6.3048 CC/G STP MAX VOL FOR PLOT = 6.3048 CC/G STP



22-87 DSS CYCLONE B NITROGEN
STATION 2 STARTED 4/ 7/87 8:30 COMPLETED 4/ 7/87 21:22

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.001078 CC/G MAX INC FOR PLOT = 0.001078 CC/G

```
                20%          40%          60%          80%         100%
              I............I............I............I............I
  14.0 *****
  16.5 ****
  19.4 ******
  22.8 **************
  26.8 ***************
  31.6 **************
  37.1 ******************
  43.7 ***********************
  51.4 *************************
  60.5 ********************************
  71.2 ******************************
  83.8 *************************
  98.6 ***********************
 116.0 *************************
 136.5 *************************
 160.6 *************************
 188.9 *************************
 222.3 ********************************
 261.6 ************************************
 307.8 ************************************
 362.1 ***********************************
 426.1 ************************************************
 501.3 ************************************
 589.9 *********************************
 694.1 ************************************
 816.6 ************************************************
 960.9 ********************************************************************
1130.6
1330.3
1565.2
1841.7
2167.0
2549.7
3000.0
```

ALBERTA RESEARCH COUNCIL

DIGISORB 2500 V3.03

105

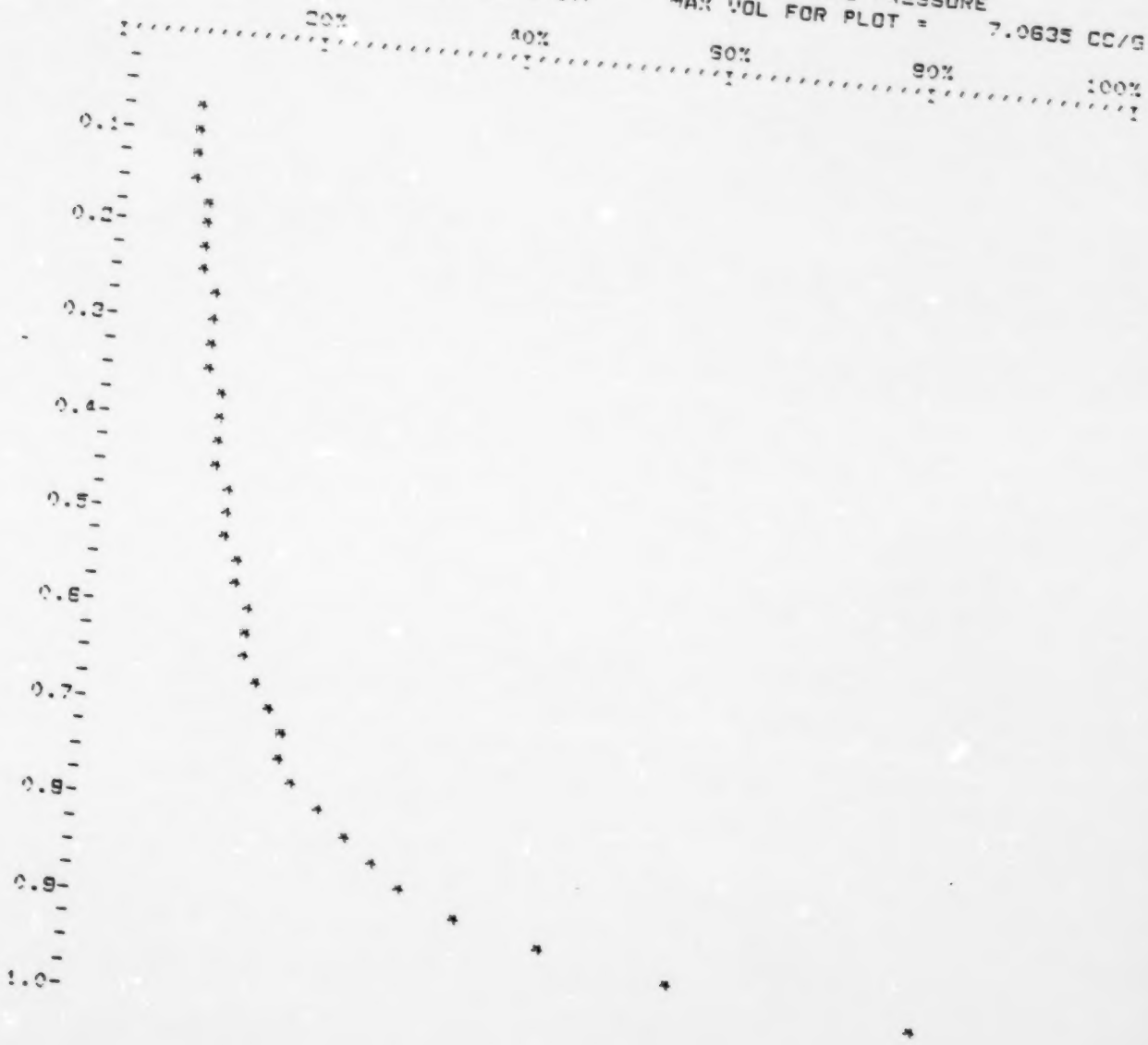
22-37 DSS CYCLONE 9 PAGE

STATION 1 STARTED 4/ 7/97 9:30 COMPLETED 4/ 7/97 23:39 NITROGEN

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE

MAX VOL SORBED = 7.0635 CC/G STP MAX VOL FOR PLOT = 7.0635 CC/G STP



ALBERTA RESEARCH COUNCIL

DIGISORB 2600 V3.03 PAGE
105

23-87 DSS CYCLONE 9 NITROGEN
STATION 4 STARTED 4/ 7/87 8:30 COMPLETED 4/ 7/87 23:39

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.001016 CC/G MAX INC FOR PLOT = 0.001016 CC/

```
                20%          40%          60%          80%         100%
     I.....I.....I.....I.....I.....I.....I.....I.....I.....I.....I
  14.0 ***
  16.5 ***
  19.4 ****
  22.8 **********
  26.8 *********
  31.6 *******
  37.1 **********
  43.7 ************
  51.4 ****************
  60.5 ******************
  71.2 **********************
  83.8 *************************
  98.6 ********************************
 116.0 ****************************************
 136.5 ***************************************
 160.6 ******************************************
 188.9 ************************************************
 222.3 ***************************************************
 261.6 ******************************************************
 307.8 ********************************************************
 362.1 ***********************************************************
 426.1 ************************************************
 501.3 ***************************************
 589.9 ***************************************
 694.1 ********************************************************
 816.6 ************************************************************
 960.9 ***************************************************************
1130.6
1330.3
1565.2
1841.7
2167.0
2549.7
3000.0
```

ALBERTA RESEARCH COUNCIL

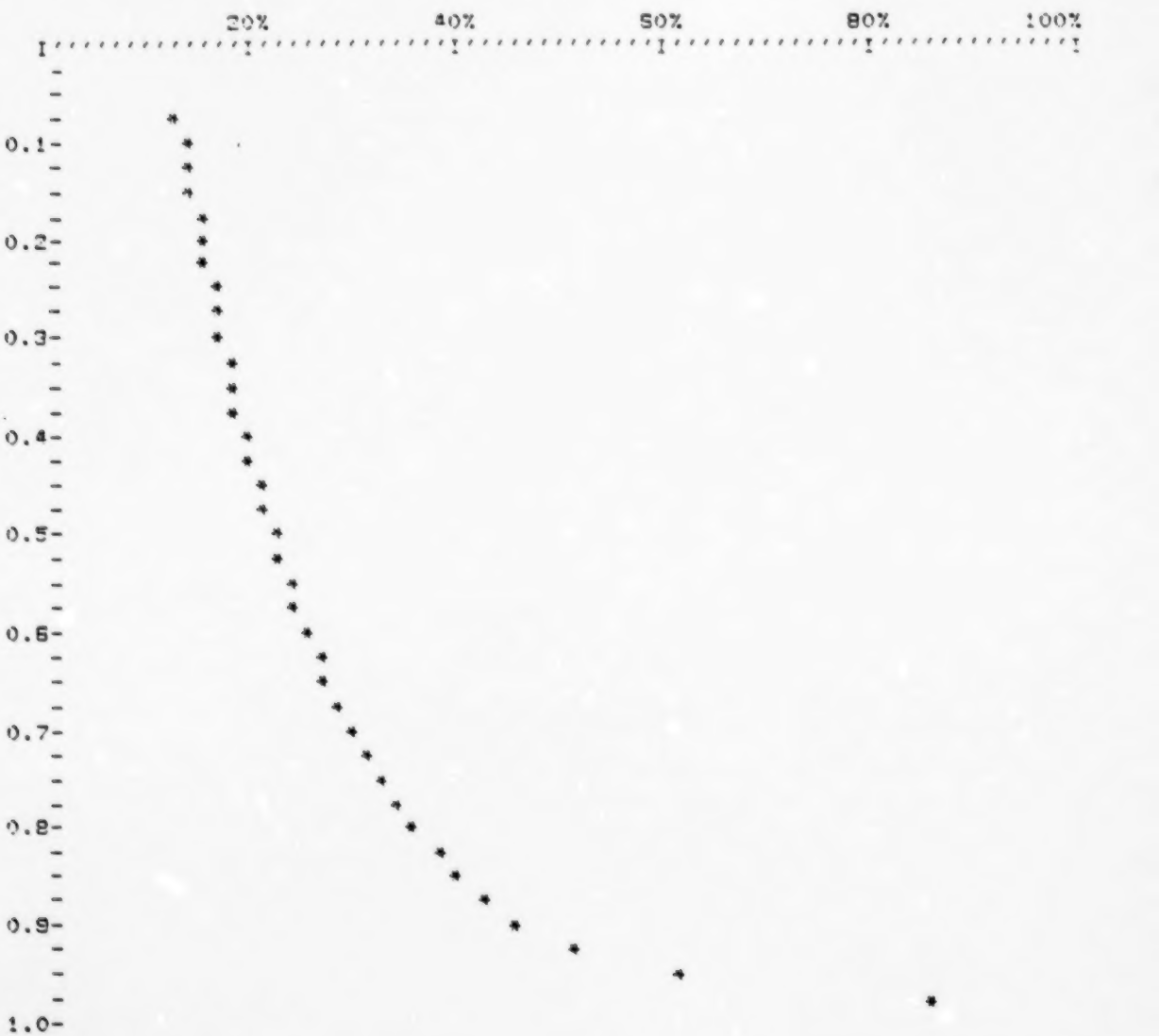
DIGISORB 2600 V3.03
105

24-87 DSS CYCLONE 10 NITROGEN
STATION 2 STARTED 4/10/87 8:23 COMPLETED 4/10/87 18: 5

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE

MAX VOL SORBED = 9.9530 CC/G STP MAX VOL FOR PLOT = 9.9530 CC/G STP



ALBERTA RESEARCH COUNCIL

DIGISORB 2600 V3.02 PAGE
105

24-87 DSS CYCLONE 10 NITROGEN
STATION 2 STARTED 4/10/87 8:23 COMPLETED 4/10/87 18: 5

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.002483 CC/G MAX INC FOR PLOT = 0.002483 CC/

```
                20%          40%          60%          80%         100%
    I'''''''''''''I''''''''''''I''''''''''''I''''''''''''I''''''''''''I
  14.0
  16.5 ******
  19.4 ****
  22.8 ********
  26.8 ************
  31.6 ************
  37.1 ************
  43.7 ***************
  51.4 ******************
  60.5 ********************
  71.2 ********************
  83.8 ****************
  98.6 **************
 116.0 ******************
 136.5 **************
 160.6 ***********
 188.9 ****************
 222.3 ************************
 261.6 ********************
 307.8 ******************
 362.1 ********************
 426.1 ********************************
 501.3 ******************
 589.9 ********************
 694.1 ********************************************
 816.6 ************************************************************************
 960.9 ************************************
1130.6
1330.3
1565.2
1841.7
2167.0
2549.7
3000.0
```

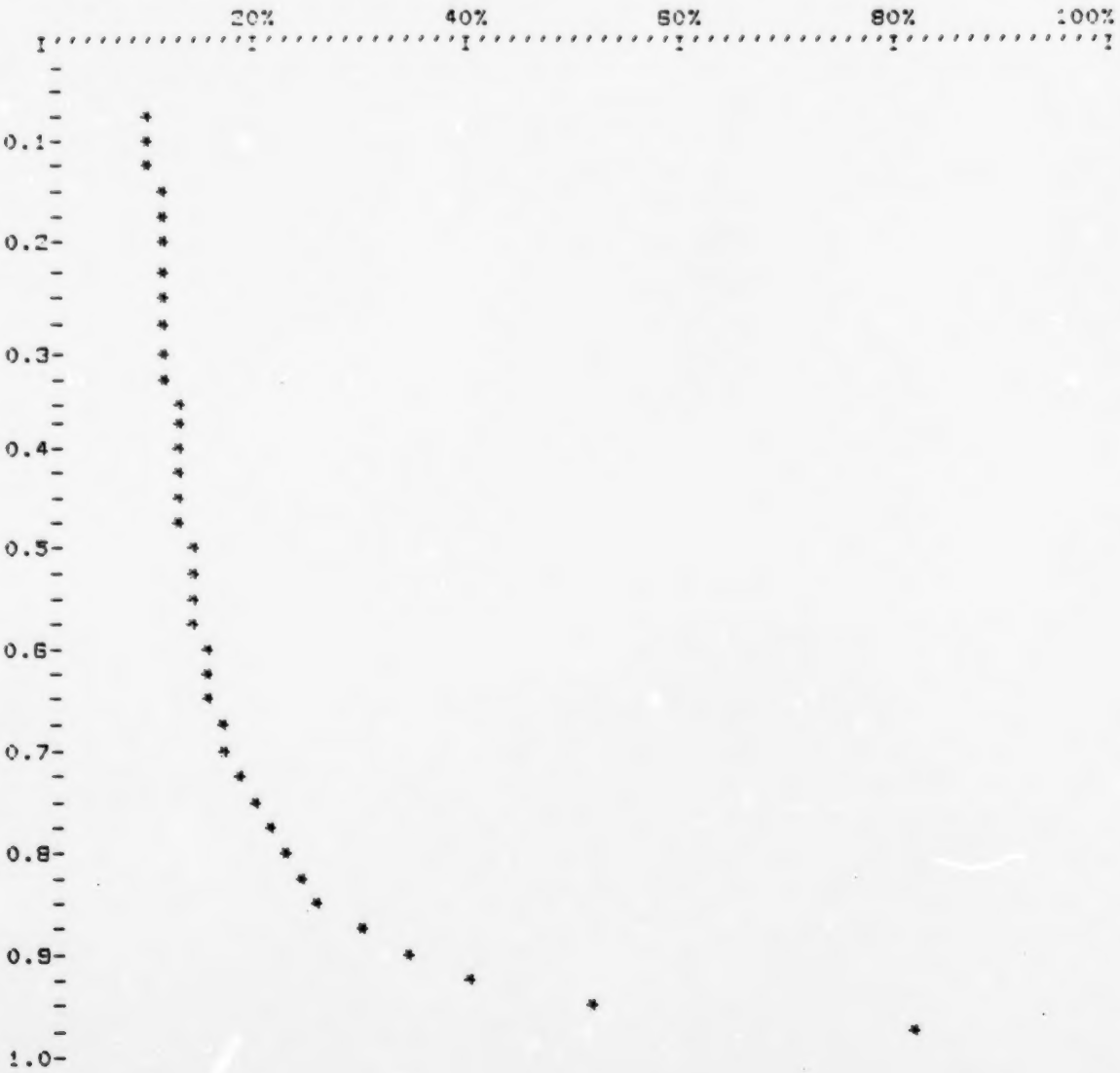
ALBERTA RESEARCH COUNCIL

DIGISORB 2600 V3.03 PAGE 1
105

25-87 DSS CYCLONE 11 NITROGEN
STATION 3 STARTED 4/10/87 8:23 COMPLETED 4/10/87 20:26

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE
MAX VOL SORBED = 5.9734 CC/G STP MAX VOL FOR PLOT = 5.9734 CC/G STP



25-87 DSS CYCLONE 11 NITROGEN
STATION 3 STARTED 4/10/87 8:23 COMPLETED 4/10/87 20:26

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.001160 CC/G MAX INC FOR PLOT = 0.001160 CC/G

```
                20%          40%          60%          80%         100%
     I.....I.....I.....I.....I.....I.....I.....I.....I.....I.....I
  14.0
  16.5 ****
  19.4 *
  22.8
  26.8
  31.6
  37.1 **
  43.7 ***
  51.4 ******
  60.5 ***********
  71.2 ************
  83.8 ***************
  98.6 ******************
 116.0 ********************
 136.5 ************************
 160.6 *************************
 188.9 ******************************
 222.3 ******************************
 261.6 ******************************
 307.8 ***********************************
 362.1 ********************************************
 425.1 ********************************************
 501.3 ***********************************
 589.9 ***********************************
 694.1 ******************************************************
 816.6 ************************************************************************
 960.9
1130.6
1330.3
1565.2
1841.7
2167.0
2549.7
3000.0
```

ALBERTA RESEARCH COUNCIL

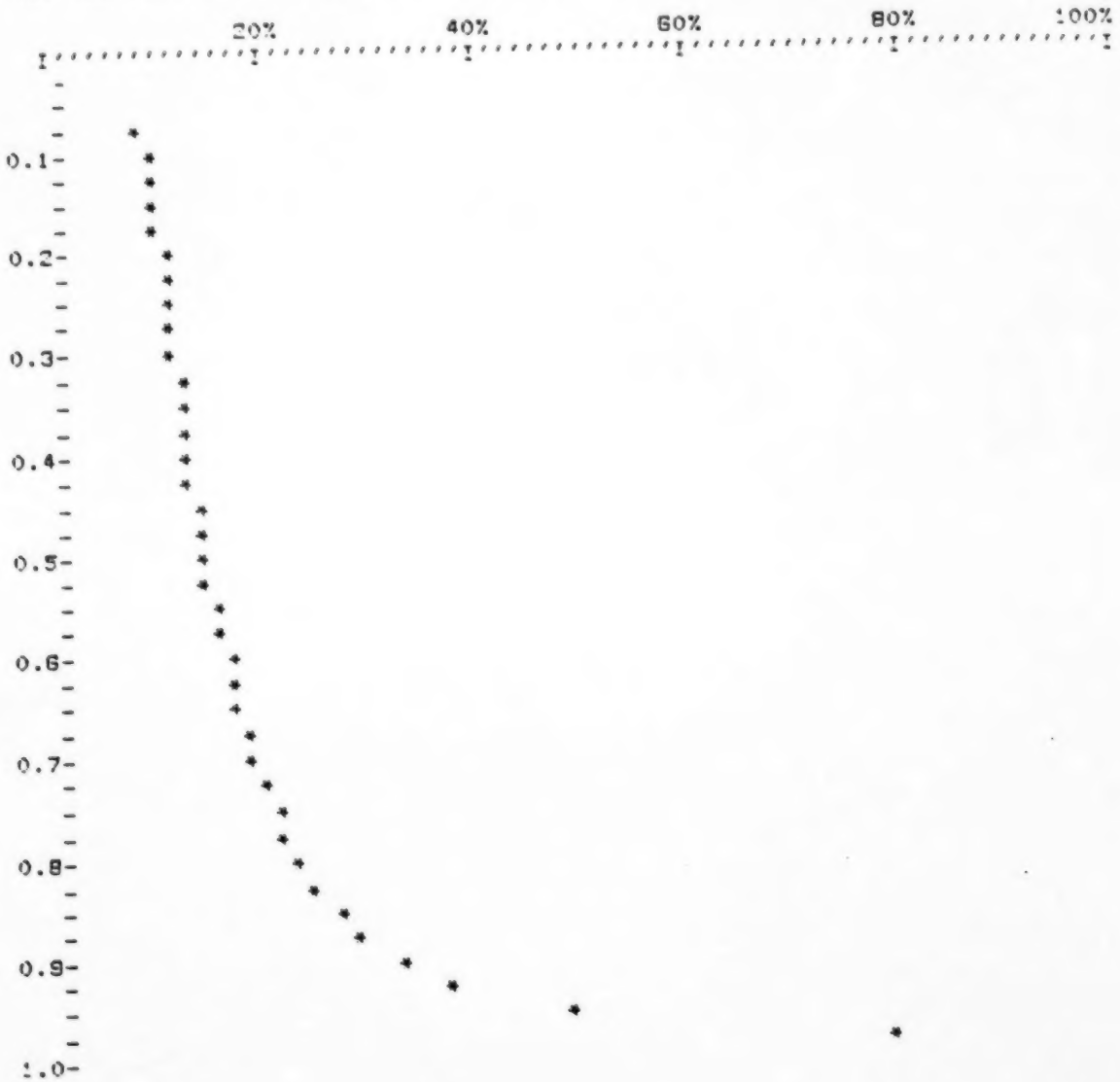
DIGISORB 2500 V3.03
105

26-97 DSS CYCLONE 12 NITROGEN
STATION 4 STARTED 4/10/97 9:23 COMPLETED 4/10/87 23:10

ISOTHERM PLOT

% OF MAXIMUM VOLUME VERSUS RELATIVE PRESSURE

MAX VOL SORBED = 8.2089 CC/G STP MAX VOL FOR PLOT = 8.2089 CC/G STP



26-87 DSS CYCLONE 12 NITROGEN
STATION 4 STARTED 4/10/87 8:23 COMPLETED 4/10/87 23:10

INCREMENTAL PORE VOLUME DISTRIBUTION (ADSORPTION)

% OF MAXIMUM PORE VOLUME VERSUS AVERAGE PORE DIAMETER, ANGSTROMS
MAX INC PORE VOLUME = 0.002526 CC/G MAX INC FOR PLOT = 0.002525 CC/G

```
                20%          40%          60%          80%         100%
     I'''''''''''I''''''''''''I''''''''''''I''''''''''''I''''''''''''I
  14.0
  16.5 ***
  19.4 **
  22.8 ***
  26.8 ****
  31.6 *****
  37.1 *****
  43.7 ******
  51.4 *******
  60.5 *******
  71.2 ********
  83.8 ********
  98.6 *********
 116.0 ***********
 136.5 **********
 160.6 **********
 188.9 ***********
 222.3 ****************
 261.6 ******************
 307.8 ********************
 362.1 *************************
 426.1 *************************
 501.3 ******************
 589.9 ******************
 694.1 ********************************
 816.6 ************************************************************
 960.9
1130.6
1330.3
1565.2
1841.7
2167.0
2549.7
3000.0
```

SURFACE CHARACTERISTICS OF CANADIAN COALS : PART 2

CO_2- ABSORPTION.

D.S.S. CONTRACT :

CYCLONE ENGINEERING SALES LTD
9719-60 Avenue
Edmonton, Alberta T6E 0C4

Subcontracted to :

ALBERTA RESEARCH COUNCIL
DEVON COAL RESEARCH CENTRE
P.O.Bag #1310
Devon, Alberta T0c 1E0

Purchase Order No 2667
Dated February 25, 1987.

Report Contact Person :

Dr Sujit K Chakrabartty
Senior Research Officer.
Coal Research Department.
Alberta Research Council

Submission Date : August 20, 1987.

Table of Content

1. Experimental Procedure
2. Graphs : adsorption plots of 12 measurements at 0^{o} and 4 measurements at $25^{o}C$.
3. Summary Table of experimental conditions, micropore capacity and surface area data.

Experimental Procedure

The standard volumetric method for adsorption measurement is used. In this method, the pressure, volume, and temperature of a given quantity of an adsorbate are measured and the number of moles present is calculated. The adsorbate is then brought into contact with the adsorbent. After equilibrium is attained with respect to pressure, the number of moles in the gas phase is again calculated. The difference between the number of moles present initially and the amount remaining after contact with the adsorbent represents the amount adsorbed.

The procedure introduced by Marsh and Siemieniewska(Fuel 44,355-367, 1965) was followed. The Polanyi-Dubinin equation is used to calculate surface areas of samples from CO_2 adsorption measured at 0 and 25°C :

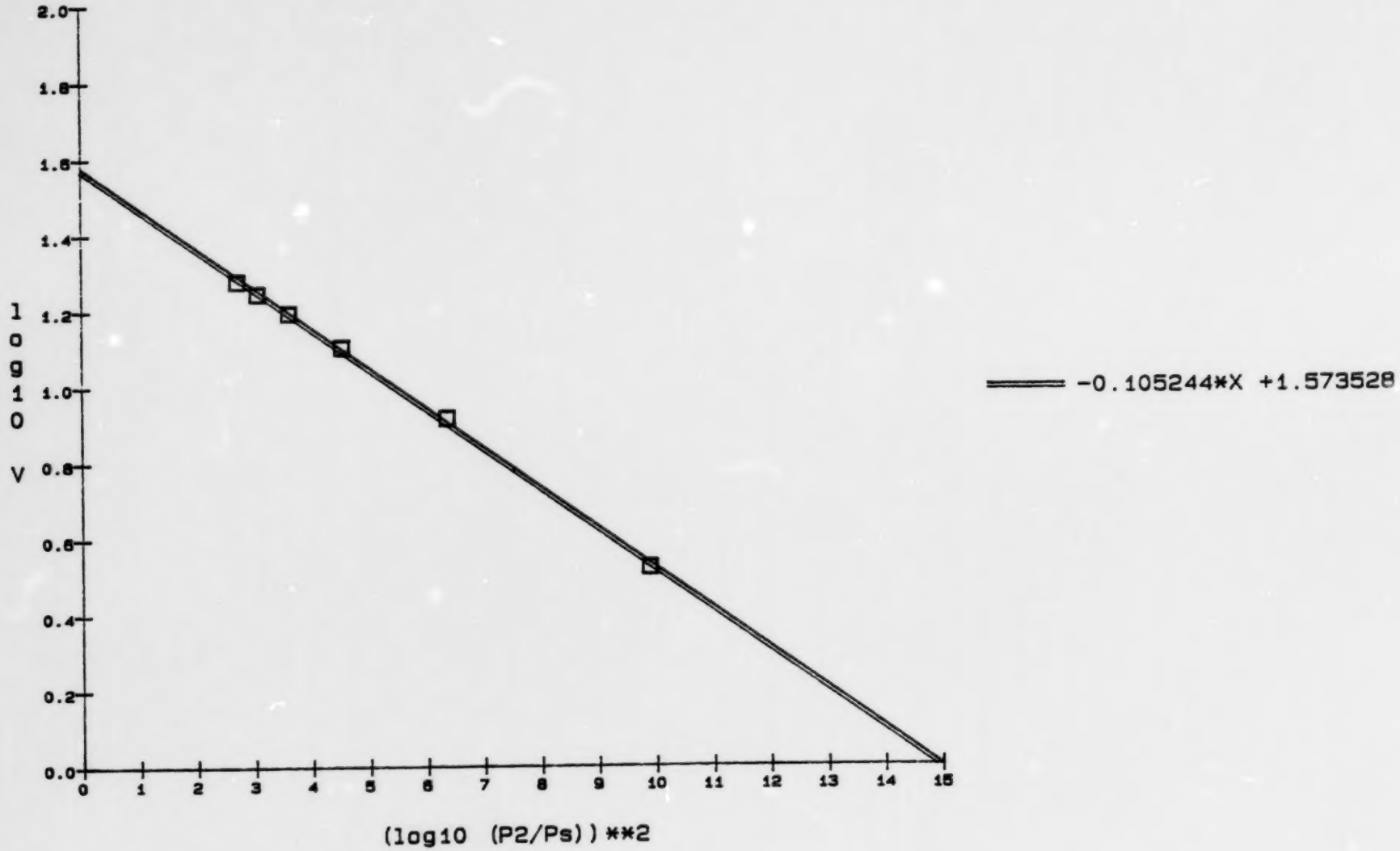
$$\log V = \log V_o - (0.434\, BT^2/b^2)\,(\log (p_s/p))^2$$

where V is the amount adsorbed at equilibrium pressure p, V_o is the micropore capacity (m.c.), p_s is the saturation vapour pressure of the adsorbate, b the affinity coefficient of the adsorbate relative to N_2 or benzene, and B a constant which is a measure of the micropore size.

The intercept of the plot between log V and$(\log(p_s/p))^2$ gives the micropore capacity V_0, which, when multiplied by the cross-sectional area of an adsorbed molecule, gives the micropore surface area. The P-D equation is usually applicable over the relative vapour pressure range of 1×10^{-5} to o.2. The unique feature of the P-D equation is that it permits the evaluation of micropore capacity and, hence, surface area from adsorption data obtained below 1 atm pressure in a conventional volumetric apparatus. Dubinin also suggested that due to an adsorption force field in the entire volume of micropores, adsorption should results in volume filling of the pores. Thus the absolute values, surface area of microporous adsorbents will depend on the assignment of a definite value for the cross-sectional area of the adsorbate molecule. Marsh used 17 (A^2) as the cross-sectional areas of adsorbed CO_2 at 0°C.

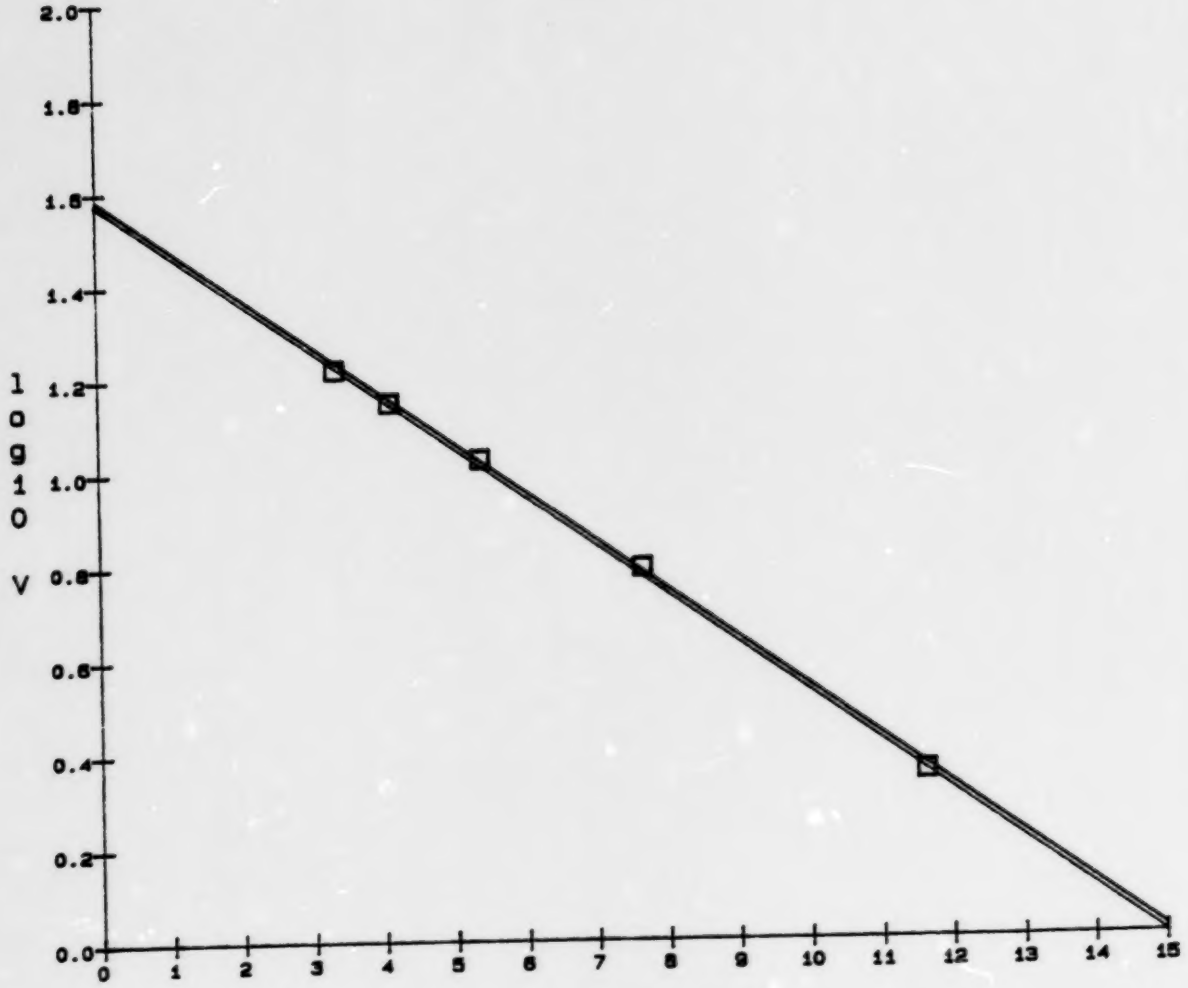
The results reported on the following pages are based on adsorption data points only. A few samples were analyzed twice to confirm the repeatability of the experiments.

15-87 CYCLONE #1 - OC RUN



Surface Area = 171.10822

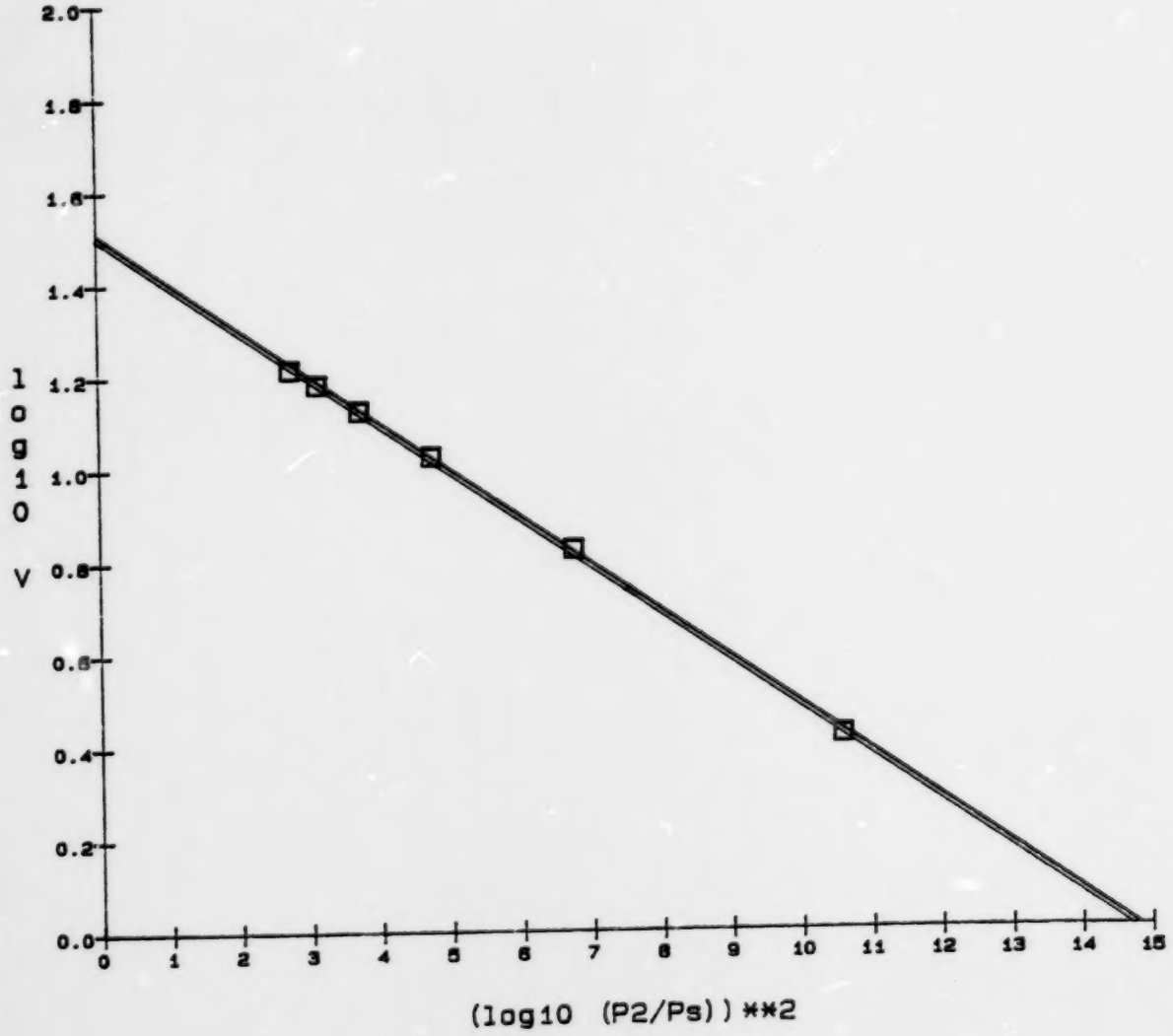
19-87 CYCLONE #5 - 0C RUN



-0.104921*X +1.581433

Surface Area = 174.250875

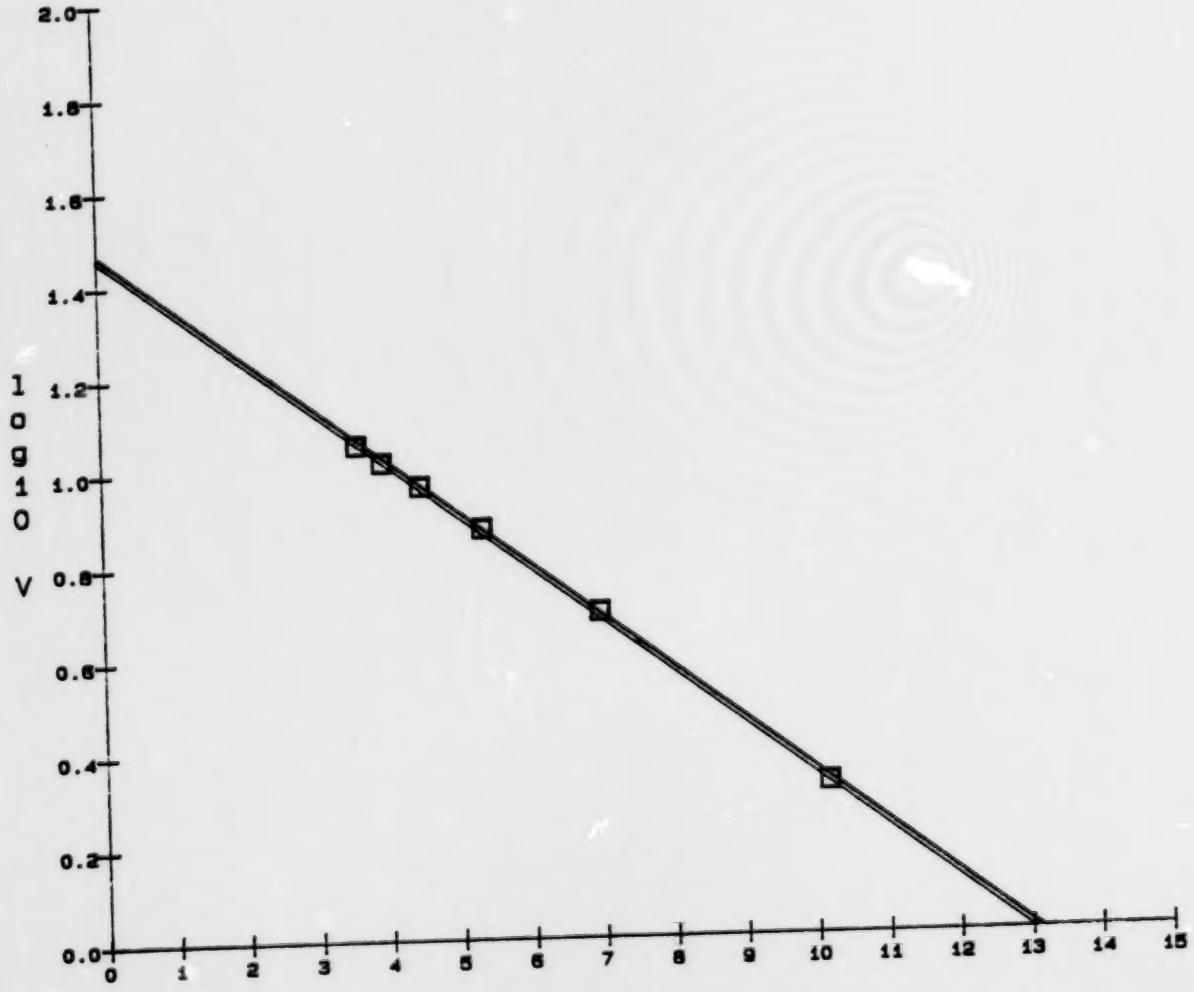
CYCLONE #9 - OC RUN



-0.101866*X +1.503821

Surface Area = 145.734925

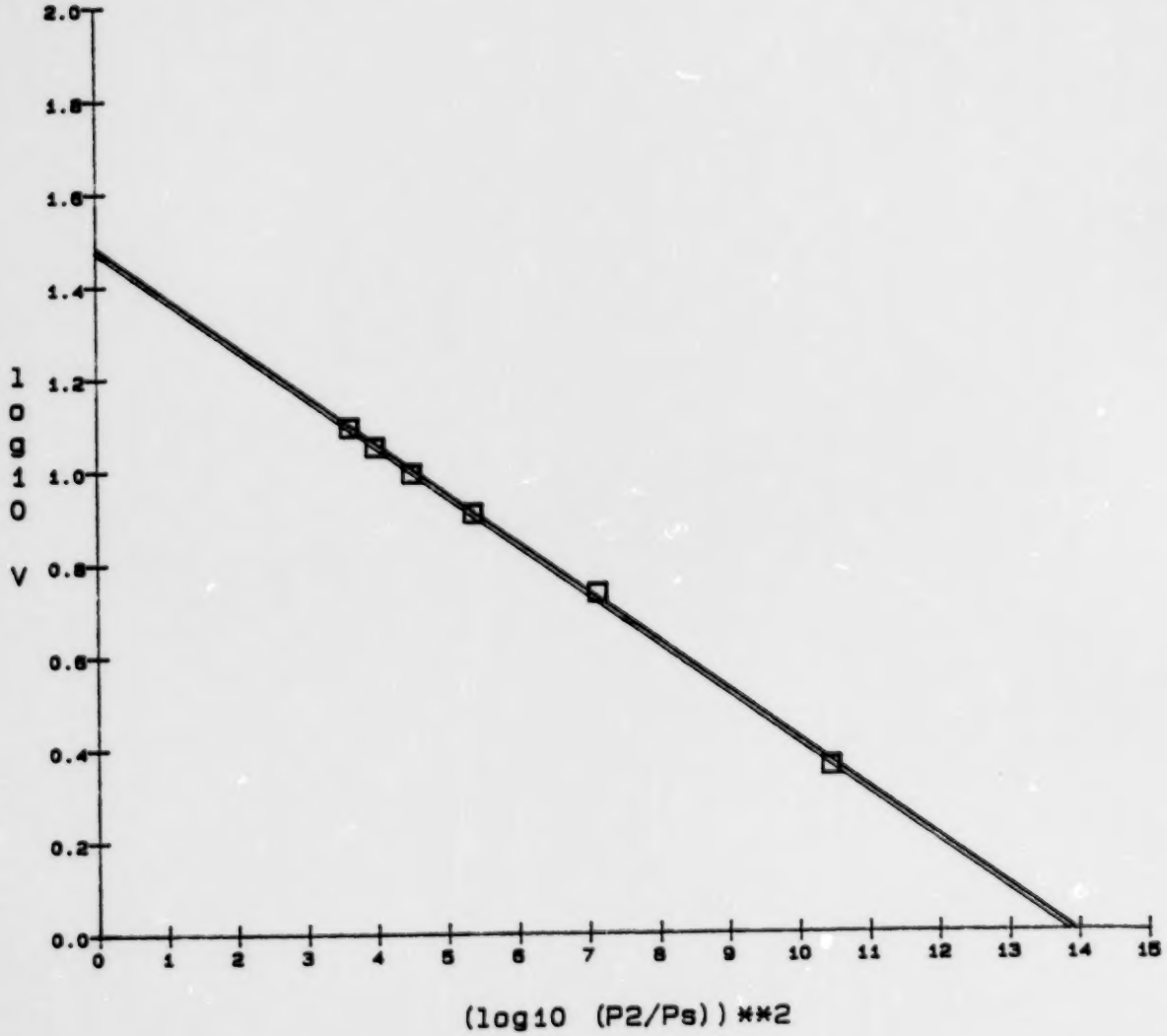
CYCLONE #9 - 25 C RUN



-0.111833*X +1.463925

Surface Area = 132.943555

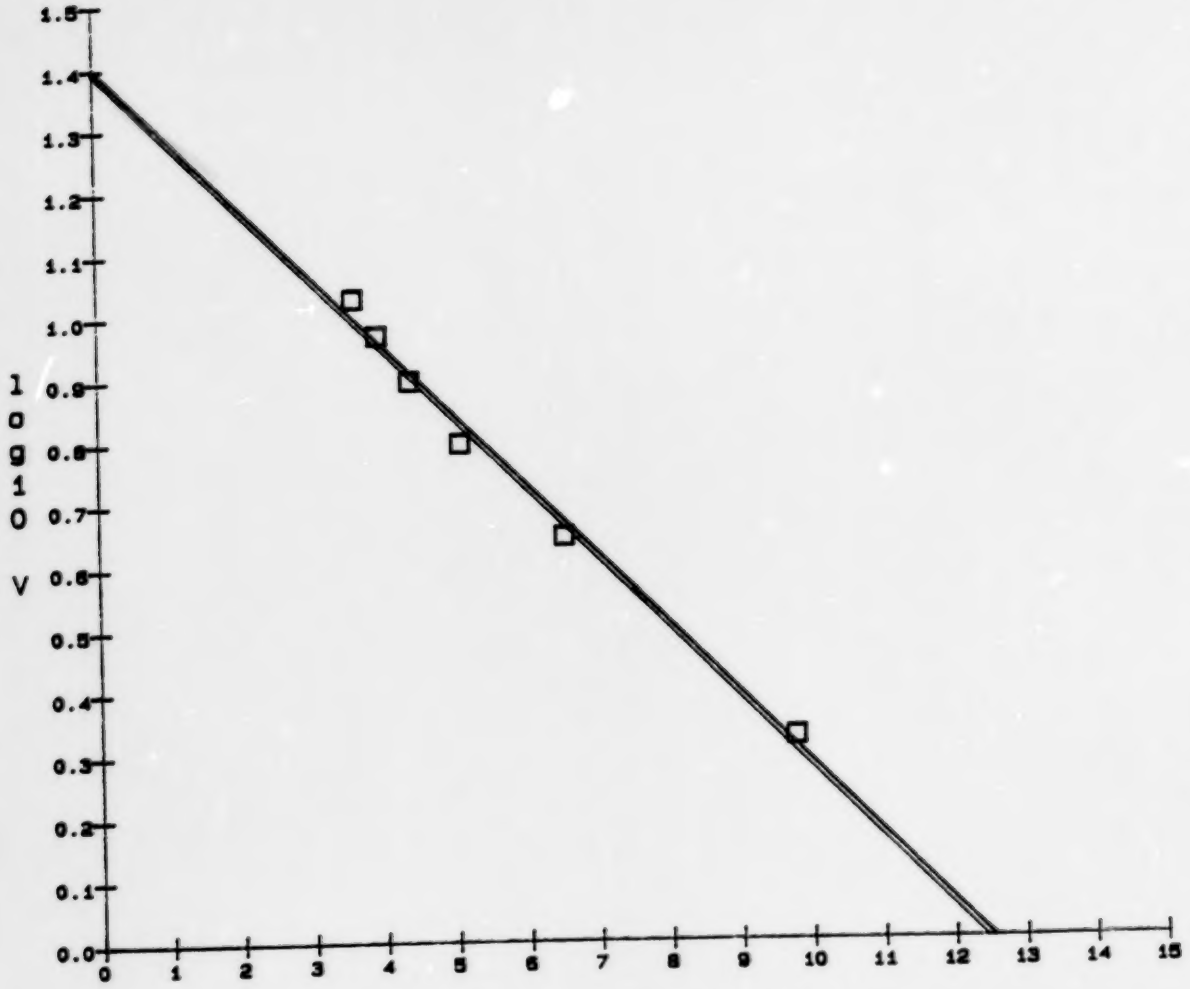
19-87 CYCLONE #5 - 25C RUN



-0.106262*X +1.478975

Surface Area = 137.631479

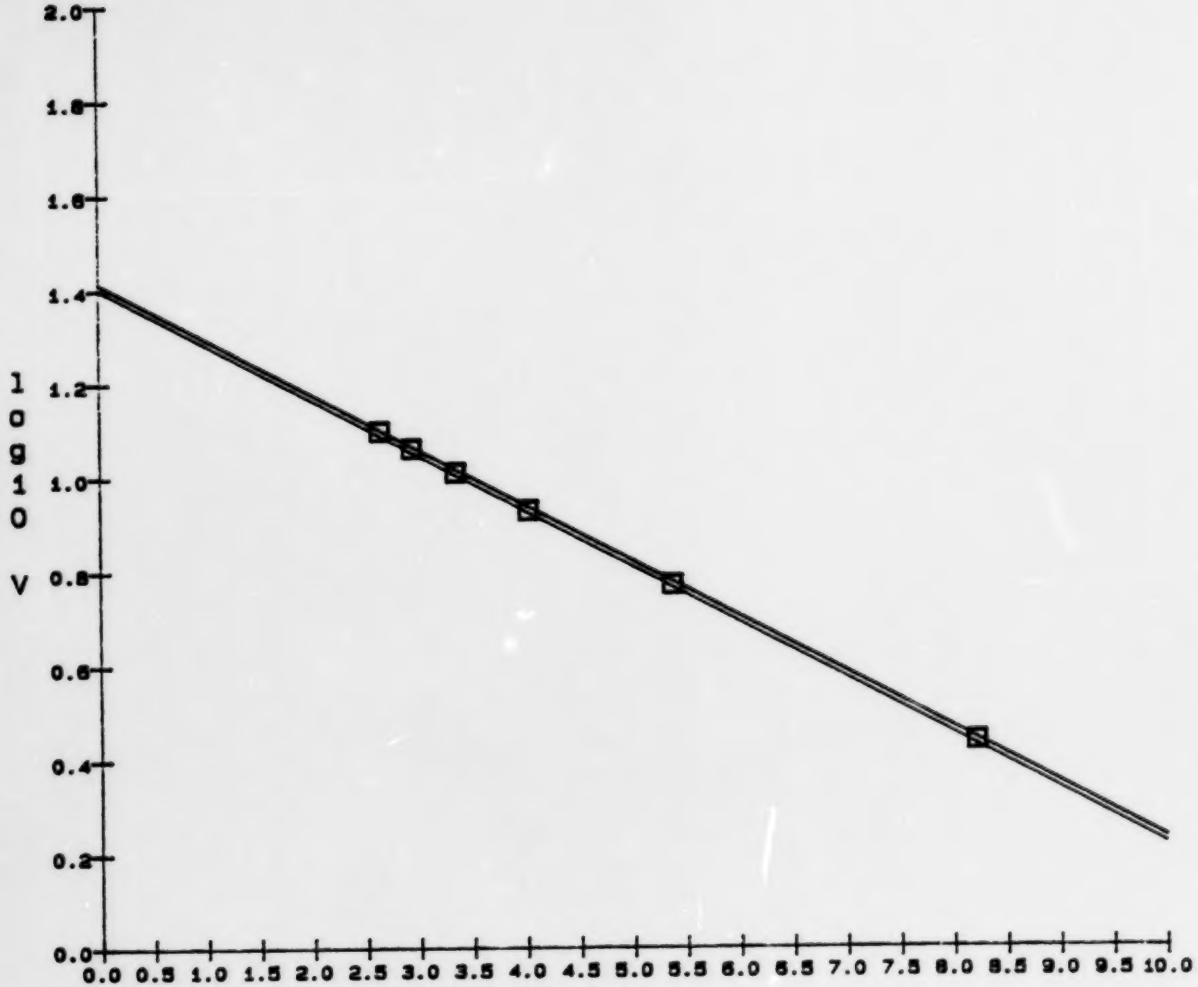
15-87 CYCLONE #1 - 25C



-0.111742*X +1.397744

Surface Area = 114.152631

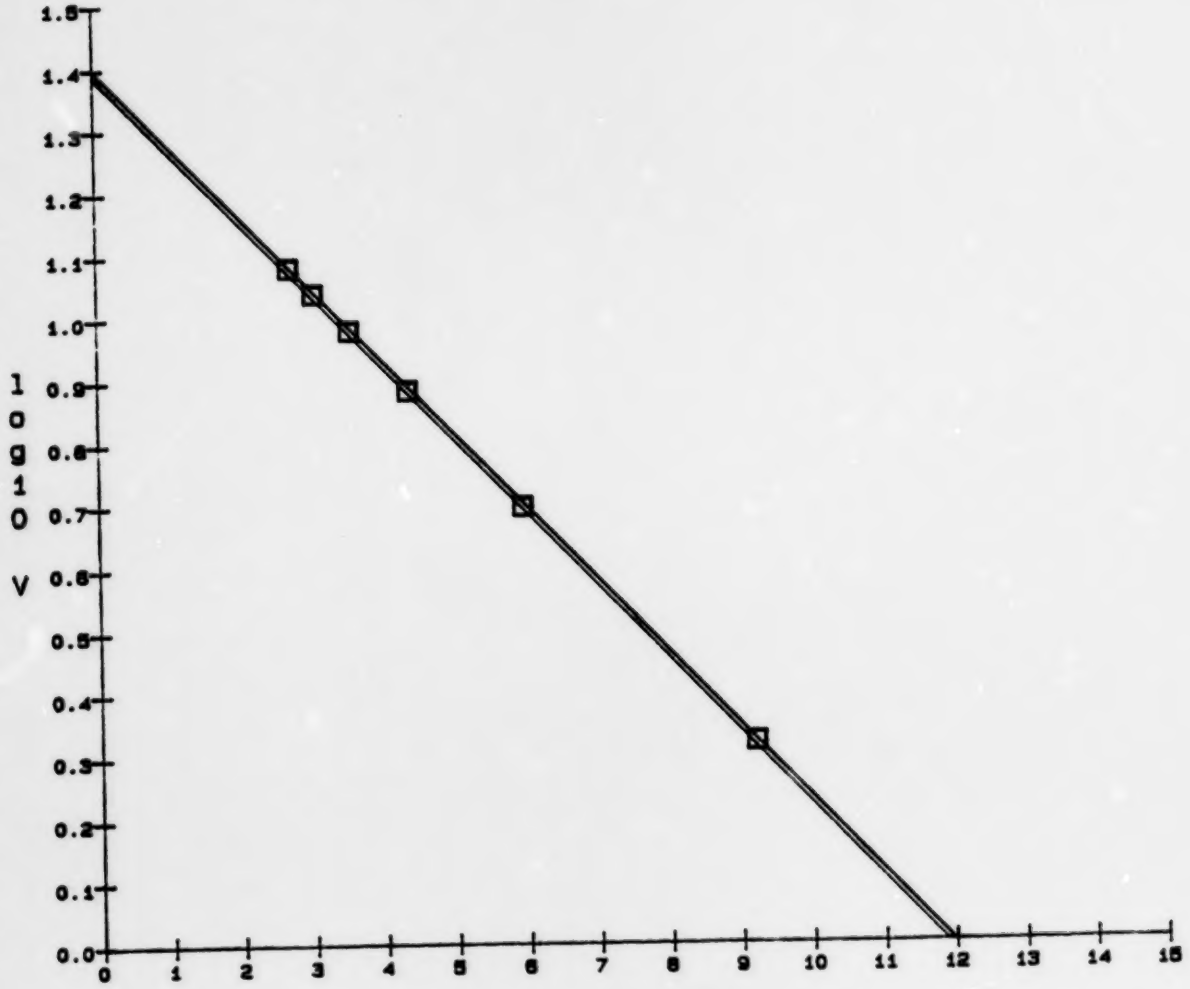
CYCLONE #2 - OC RUN



-0.118165*X +1.407531

Surface Area = 116.754574

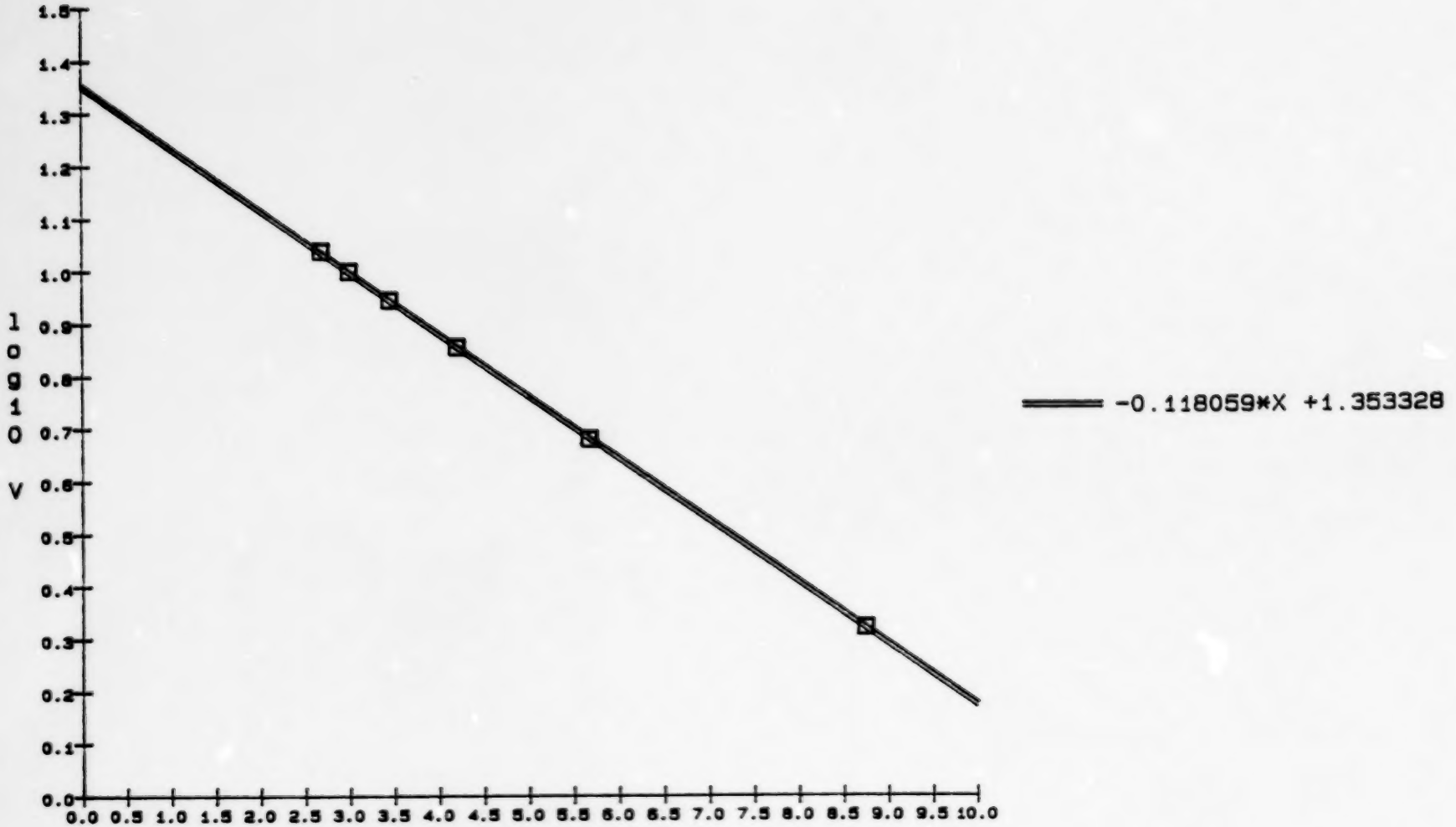
CYCLONE #6 - OC RUN



-0.116824*X +1.392119

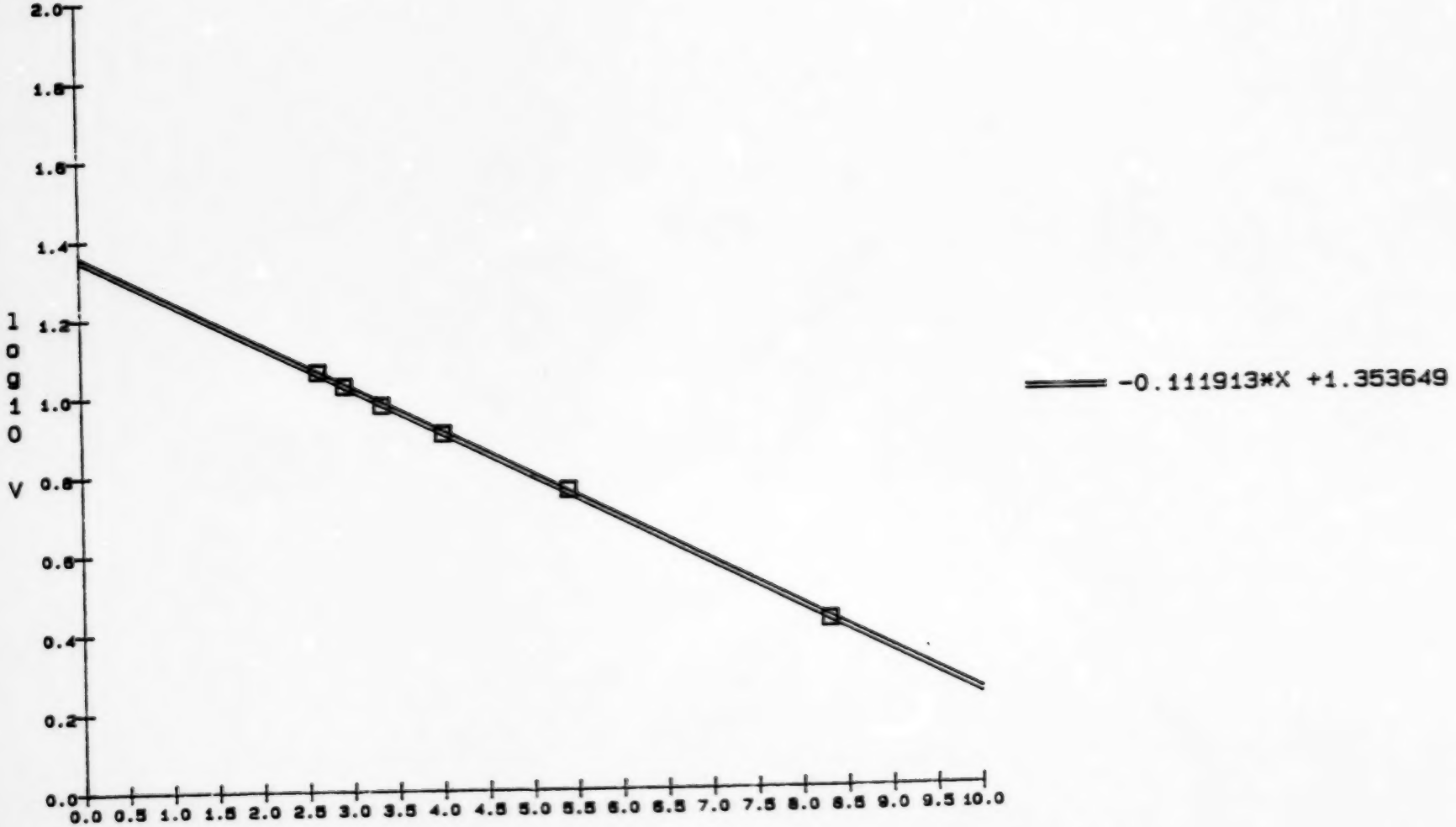
Surface Area = 112.683729

CYCLONE#10 - OC RUN



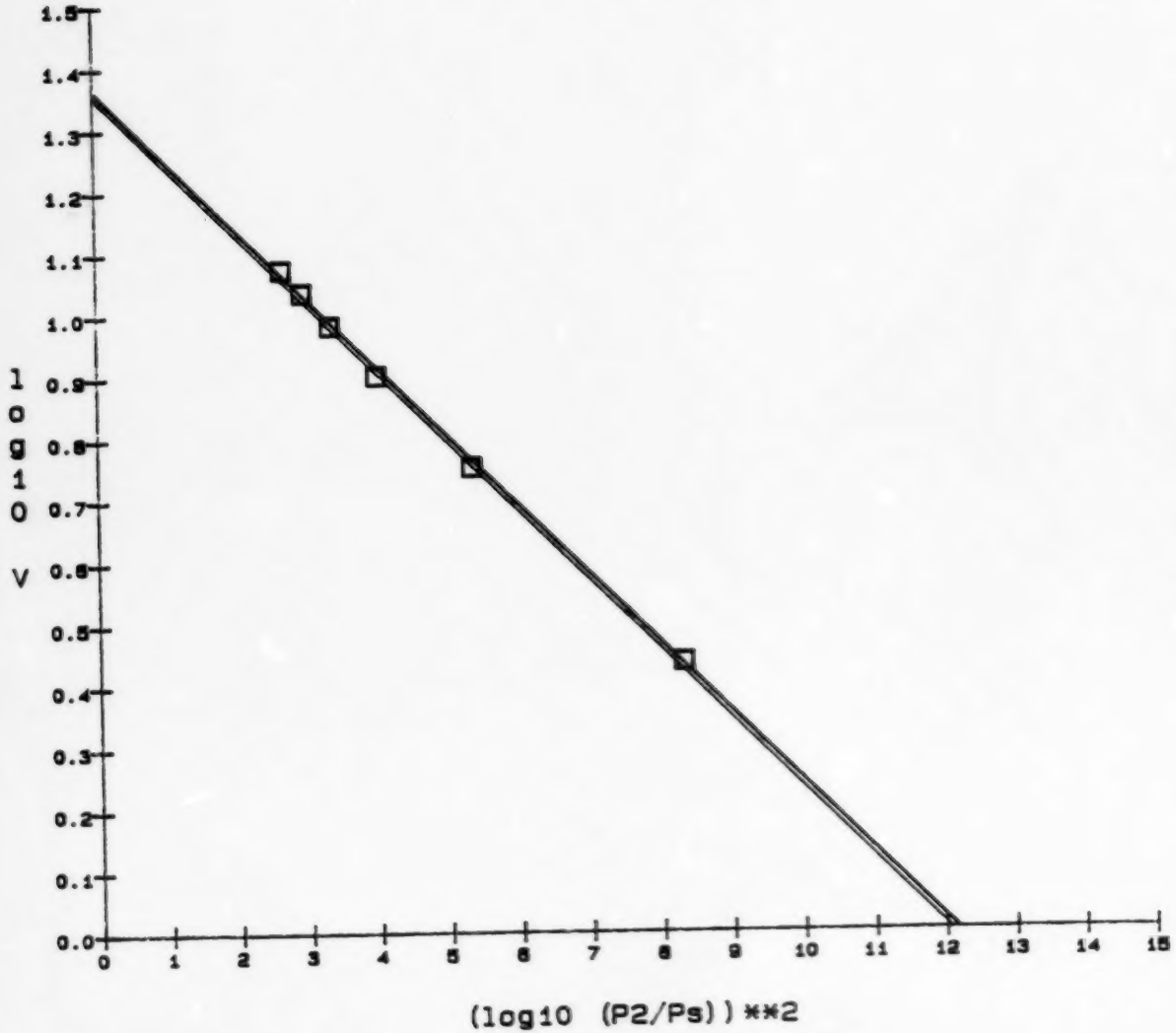
Surface Area = 103.055355

17-87 CYLONE #3 - 0C RUN



Surface Area = 103.131476

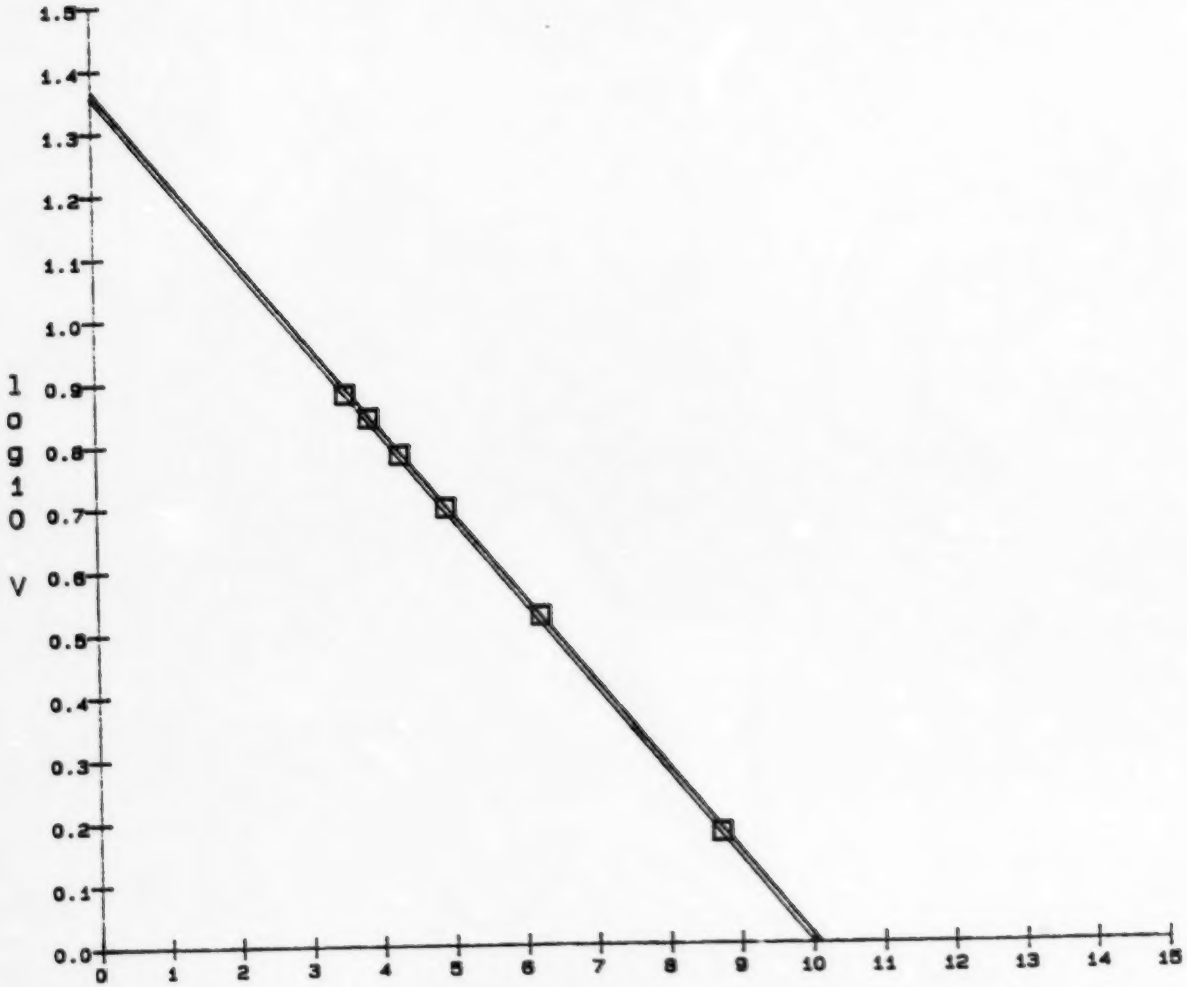
CYCLONE #3 - 0C RUN (REPEAT)



-0.112143*X +1.358935

Surface Area = 104.394534

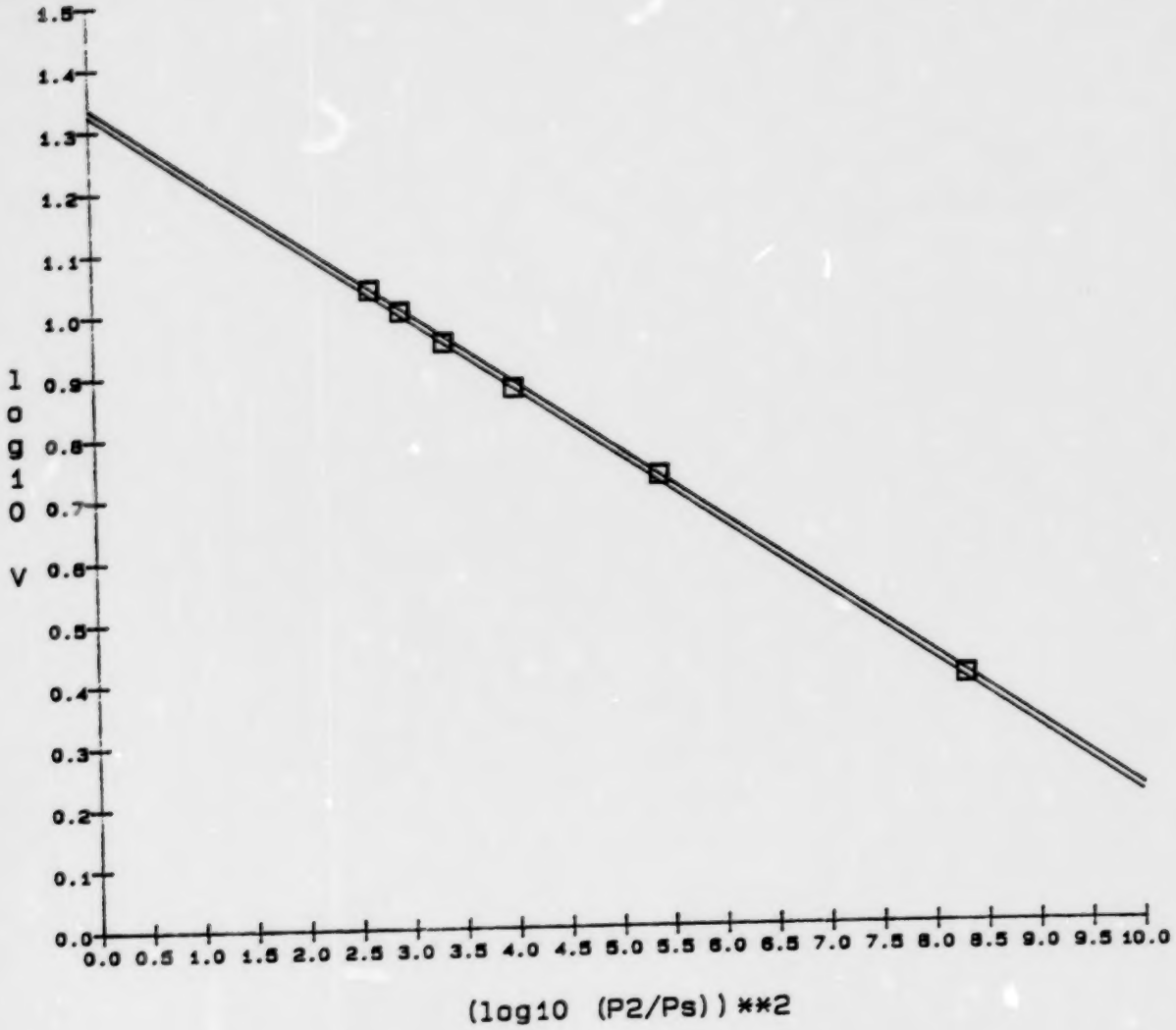
17-87 CYCLONE #3 - 25C



-0.135484*X +1.362574

Surface Area = 105.272926

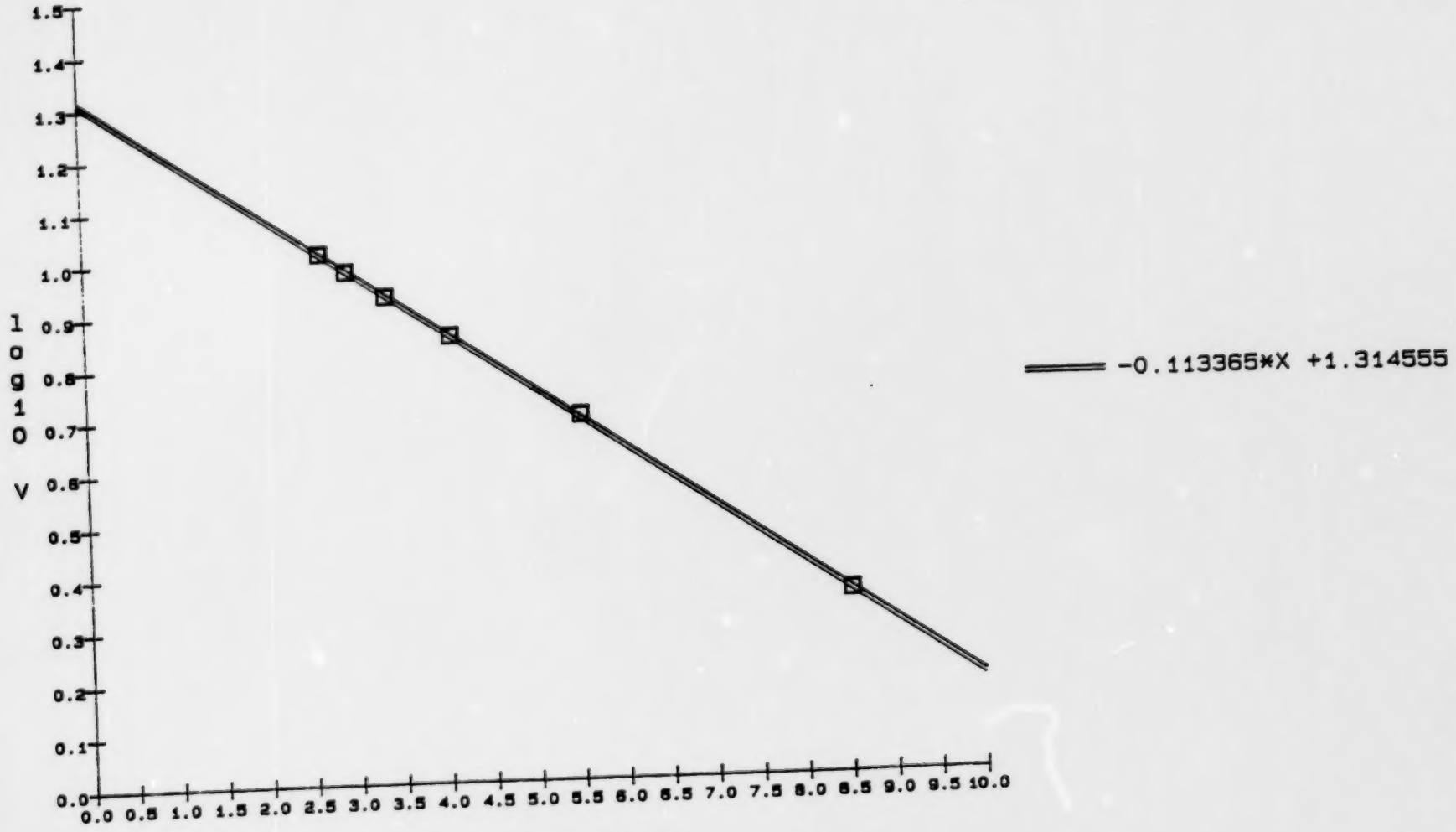
CYCLONE #7 - OC RUN



-0.111664*X +1.330142

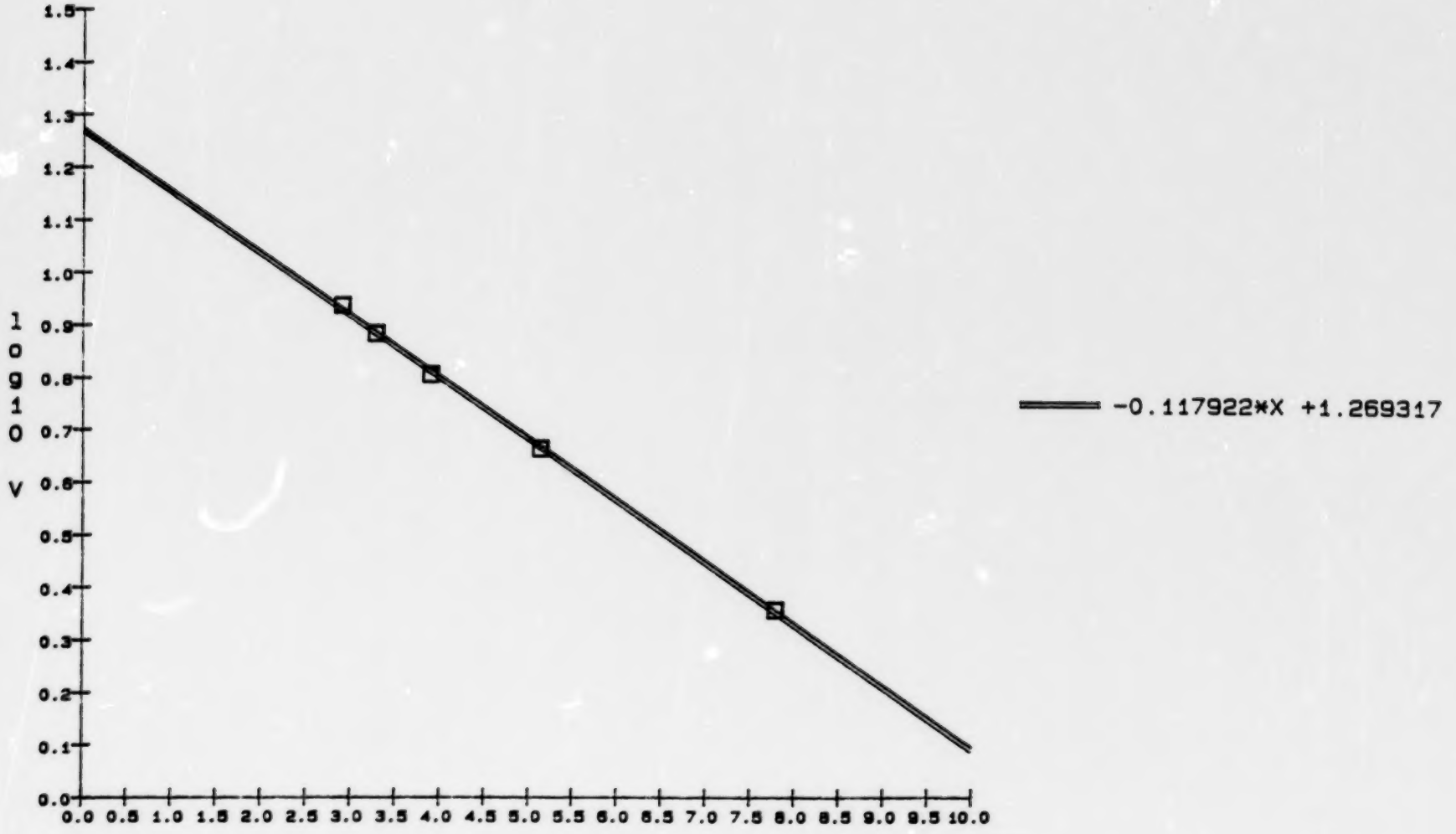
Surface Area = 97.697684

CYCLONE #11 - OC RUN



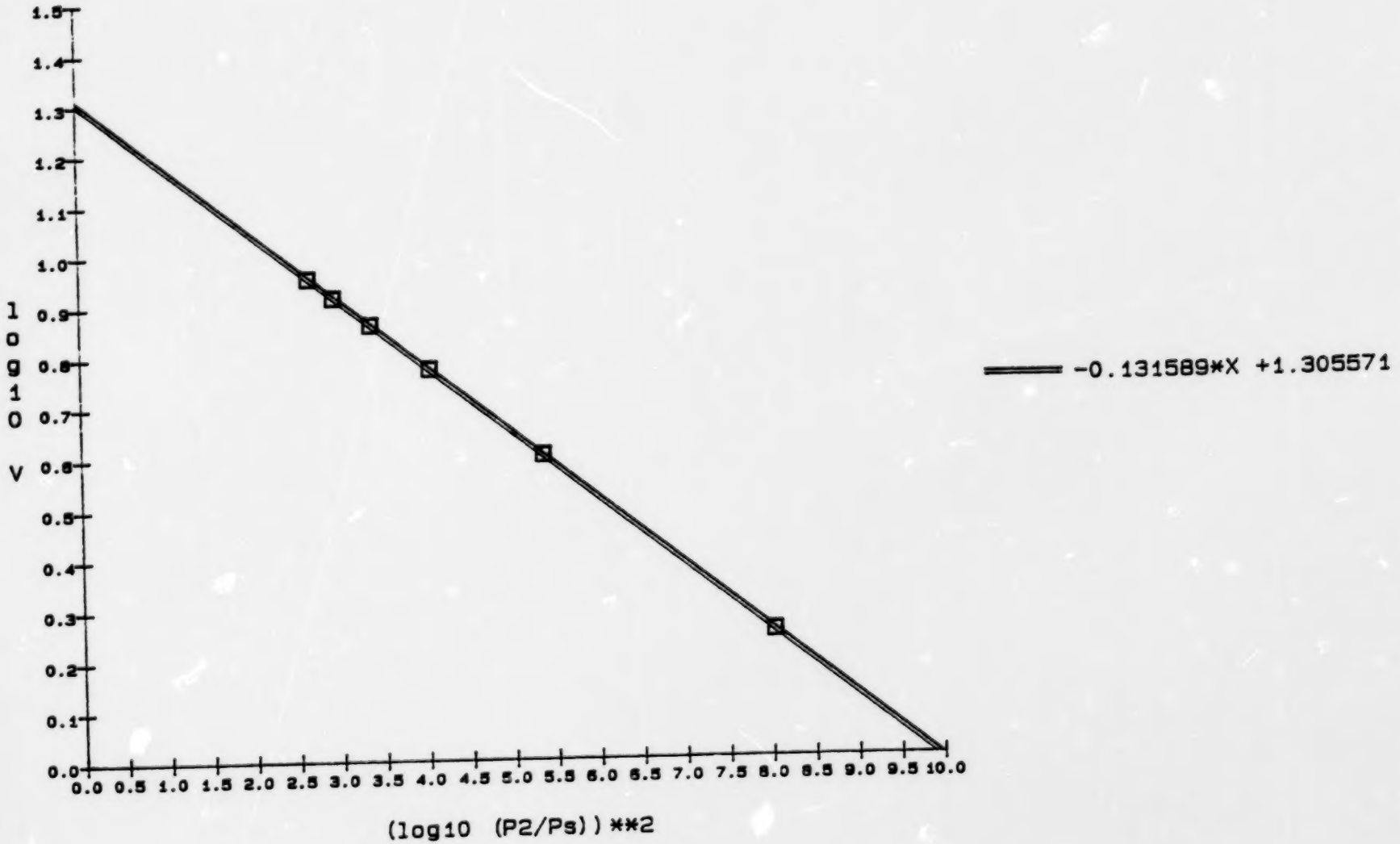
Surface Area = 94.25345

CYCLONE #4 - OCRUN



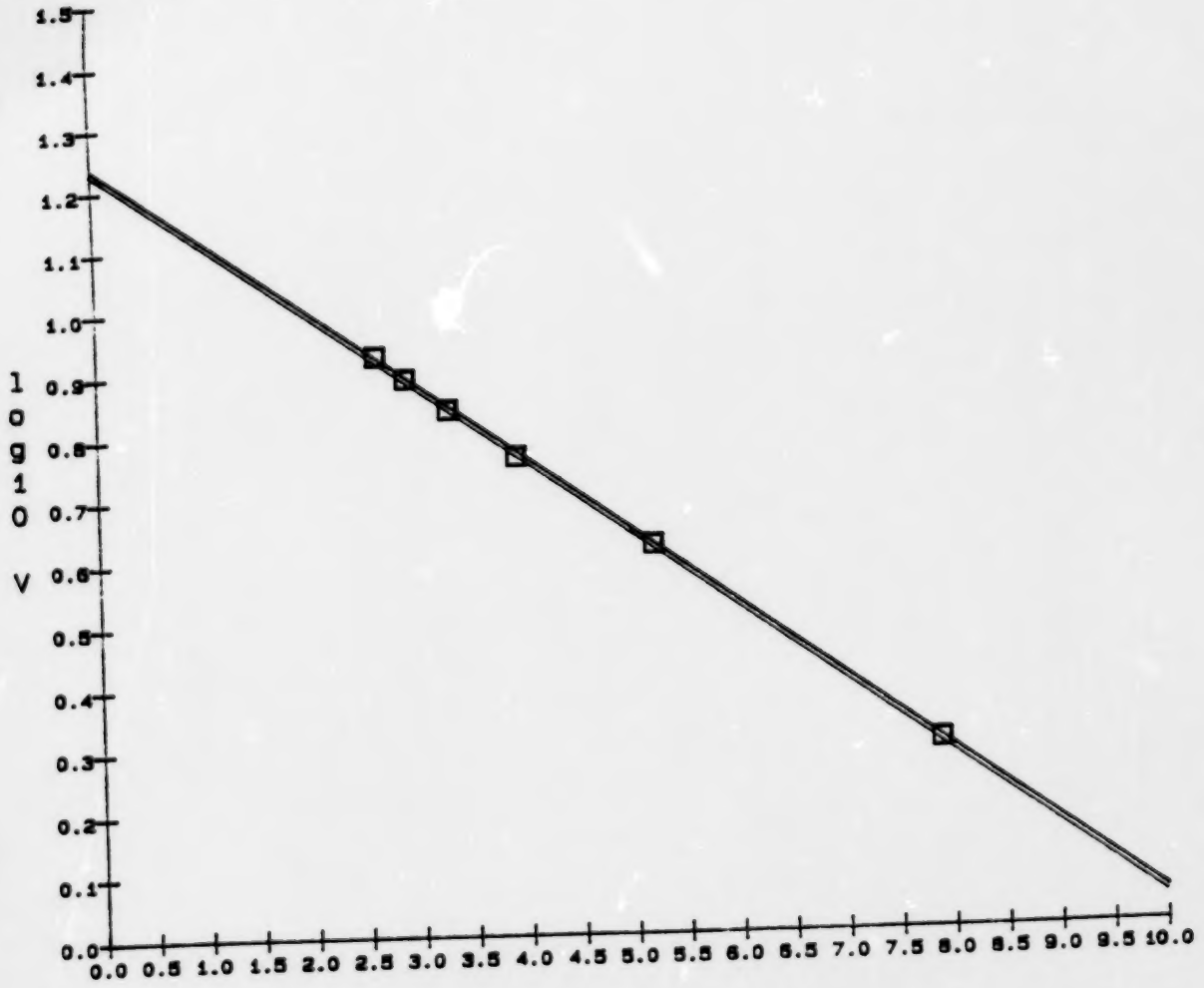
Surface Area = 84.929684

CYCLONE #8 - OC RUN



Surface Area = 92.32367

CYCLONE #12 - OC RUN



-0.118655*X +1.234592

Surface Area = 78.403354

SUMMARY TABLE : EXPERIMENTAL CONDITIONS & ABSORPTION DATA

Sample I.D.	Vacuum Pre-heating		Degasing		Absorption						
	T °C	Hr	T °C	Hr	T °C	t-min	sample weight, g.	Slope	y-Intercept	Micropore Capacity	Surface Area.
Dss/Cyclone											
#1	105	18	107	18	0	30	1.0951	-0.105	1.574	37.457	171.1
#5	105	18	107	18	0	30	1.8495	-0.105	1.581	38.145	174.2
#9	106	18	108	18	0	30	1.4929	-0.102	1.504	31.902	145.7
#9	106	18	111	18	25	30	1.4397	-0.112	1.464	29.102	132.9
#5	105	18	104	18	25	30	1.3855	-0.106	1.479	30.128	139.6
#1	105	18	103	18	25	30	1.2957	-0.112	1.390	24.989	114.1
#2	110	18	106	18	0	30	0.9803	-0.118	1.408	25.558	116.8
#6	106	18	110	18	0	30	1.6160	-0.117	1.392	24.667	112.7
#10	108	18	112	18	0	30	1.4651	-0.118	1.353	22.560	103.0
#3	105	18	109	18	0	30	1.0450	-0.112	1.354	22.576	103.1
#3	106	18	112	18	0	30	1.0134	-0.112	1.359	22.853	104.4
#3	105	18	102	18	25	30	1.1618	-0.135	1.363	23.045	105.3
#7	108	18	110	18	0	30	1.0947	-0.112	1.330	21.387	97.7
#11	108	18	110	18	0	30	1.3175	-0.113	1.315	20.633	94.3
#4	110	18	107	18	0	30	1.0125	-0.118	1.269	18.592	84.9
#8	108	18	115	18	0	30	1.4171	-0.132	1.306	20.210	92.3
#12	108	18	112	18	0	30	1.2089	-0.119	1.234	17.163	78.4